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(54) Title: **CURABLE RESIN COMPOSITION FOR COATING OPTICAL FIBERS AND CURED PRODUCT THEREOF**

(57) Abstract: The invention relates to a curable resin composition for coating optical fibers comprising: (1) 50-70 wt.% of a urethane (meth)acrylate obtained by reacting (A) a polyol compound, (B) a polyisocyanate compound, and (C) a (meth)acrylate compound containing a hydroxyl group; (2) a reactive diluent copolymerizable with the component (1); and (3) a polymerization initiator; wherein the rate of dimensional change of the cured product of the composition after immersion in mineral oil at 25 °C for one week is 2.5 % or less, or when forming a product with a three-layer structure, specifically, secondary layer-primary layer-secondary layer, or a five-layer structure, specifically, coloring layer-secondary layer-primary layer-secondary layer-coloring layer, using the cured product of the composition as the primary layer, the primary layer exhibits no foam defects after immersing the product in hot water at 60 °C for one week.



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CURABLE RESIN COMPOSITION FOR COATING OPTICAL FIBERS AND CURED  
PRODUCT THEREOF

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The present invention relates to a curable resin composition excelling in jelly resistance and water resistance, which is suitably used as a coating material for optical fibers, optical fiber ribbons, and the like, and to the cured product of the composition.

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In the fabrication of optical fibers, resin coating is applied over glass fibers produced by spinning molten glass for protection and reinforcement. As the resin coating, a structure in which a primary flexible coating layer is formed over the surface of the glass fiber, and a secondary rigid coating layer is formed over the primary coating is known. Glass fibers provided with the primary and secondary coating layers are called optical fibers. Optical fiber ribbons produced by placing a plurality of optical fibers provided with the resin coating side by side on a plane and bundling the optical fibers using a bundling material are also known. A resin composition for forming the primary coating layer is called a primary material. A resin composition for forming the secondary coating layer is called a secondary material. A resin composition used as the bundling material for optical fiber ribbons is called a ribbon matrix material. As the resin coating method, a method of applying a curable resin composition and curing the composition using heat or light, in particular, ultraviolet rays is widely used.

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When lateral pressure is applied to optical fibers or optical fiber ribbons from inside the cable, such as a case where optical fiber cables are bent, an optical transmission loss occurs. It is known in the art that optical fiber cables are filled with a jelly mixture in order to prevent optical transmission loss due to lateral pressure. This jelly mixture is called a jelly. When optical fibers coated with a conventional curable resin composition for coating optical fibers are used in the cable filled with the jelly, separation may occur at the interface between the glass fiber and the curable resin composition, thereby decreasing the strength of the optical fibers.

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When optical fibers coated with a conventional curable resin composition for coating optical fibers are immersed in water, an optical transmission loss may increase due to separation of the curable resin composition or formation of foams.

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In order to develop a curable resin composition capable of solving these problems, it is necessary to evaluate the jelly resistance and water resistance. However, conventional evaluation methods use optical fibers fabricated in practice. This makes the evaluation procedure troublesome and requires a long period of time.

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#### Means for Solving the Problems

The present inventors have examined an evaluation method using a sample cured product without fabricating optical fibers in practice. As a result, the present inventors have found that jelly resistance and water resistance can be  
10 evaluated in a short period of time with good reproducibility by respectively using a mineral oil immersion method and a hot water immersion method using a laminated cured layer. As a result of further examination, the present inventors have found that a curable resin composition comprising a specific amount of a urethane (meth)acrylate, a reactive diluent, and a polymerization initiator, the cured product of which exhibits a  
15 specific range of performance in these evaluation methods, exhibits excellent jelly resistance and/or water resistance and is useful for coating optical fibers. This finding has led to the completion of the present invention.

Specifically, the present invention provides a curable resin composition for coating optical fibers comprising: (1) 50-70 wt% of a urethane  
20 (meth)acrylate obtained by reacting (A) a polyol compound, (B) a polyisocyanate compound, and (C) a (meth)acrylate compound containing a hydroxyl group; (2) a reactive diluent copolymerizable with the component (1); and (3) a polymerization initiator; wherein the rate of dimensional change of the cured product of the composition after immersion in mineral oil at 25°C for one week is 2.5% or less.

25 The present invention also provides a curable resin composition for coating optical fibers comprising: (1) 50-70 wt% of a urethane (meth)acrylate obtained by reacting (A) a polyol compound, (B) a polyisocyanate compound, and (C) a (meth)acrylate compound containing a hydroxyl group; (2) a reactive diluent copolymerizable with the component (1); and (3) a polymerization initiator; wherein,  
30 when forming a product with a three-layer structure, specifically, secondary layer-primary layer-secondary layer, or a five-layer structure, specifically, coloring layer-secondary layer-primary layer-secondary layer-coloring layer, using the cured product of the composition as the primary layer, the primary layer exhibits no foam defects after immersing the product in hot water at 60°C for one week.

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### Brief Description of the Drawings

Figure 1 is a perspective view showing a five-layer cured product.

Figure 2 is a perspective view showing a five-layer cured product.

Figure 3 is a perspective view showing a three-layer cured product.

5 Figure 4 is a perspective view showing a three-layer cured product.

### Preferred Embodiments of the Invention

The curable resin composition for coating optical fibers of the present invention comprises a urethane (meth)acrylate as the component (1) obtained by  
10 reacting (A) a polyol compound, (B) a polyisocyanate compound, and (C) a (meth)acrylate compound containing a hydroxyl group

As specific examples of the method of preparing the urethane (meth)acrylate (1), a method of reacting the polyol (A), polyisocyanate compound (B), and hydroxyl group-containing (meth)acrylate (C) all together; a method of reacting the  
15 polyol (A) and the polyisocyanate compound (B), and reacting the resulting product with the hydroxyl group-containing (meth)acrylate (C); a method of reacting the polyisocyanate compound (B) and the hydroxyl group-containing (meth)acrylate (C), and reacting the resulting product with the polyol (A); a method of reacting the polyisocyanate compound (B) and the hydroxyl group-containing (meth)acrylate (C),  
20 reacting the resulting product with the polyol (A), and reacting the resulting product with hydroxyl group-containing (meth)acrylate (C); and the like can be given.

As examples of the polyol (A), polyether diols obtained by ring-opening polymerization of one ion-polymerizable cyclic compound such as polyethylene glycol, polypropylene glycol, polytetramethylene glycol,  
25 polyhexamethylene glycol, polyheptamethylene glycol, and polydecamethylene glycol, polyether diols obtained by ring-opening copolymerization of two or more ion-polymerizable cyclic compounds, and the like can be given. As examples of ion-polymerizable cyclic compounds, cyclic ethers such as ethylene oxide, propylene oxide, butene-1-oxide, isobutene oxide, oxetane, 3,3-dimethyloxetane, 3,3-bischloromethyloxetane, tetrahydrofuran, 2-methyltetrahydrofuran, 3-methyltetrahydrofuran, dioxane, trioxane, tetraoxane, cyclohexene oxide, styrene oxide, epichlorohydrin, glycidyl methacrylate, allyl glycidyl ether, allyl glycidyl carbonate, butadiene monoxide, isoprene monoxide, vinyloxetane, vinyltetrahydrofuran, vinylcyclohexene oxide, phenyl glycidyl ether, butyl glycidyl ether,  
30 and glycidyl benzoate can be given. Polyether diols obtained by the ring-opening  
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copolymerization of these ion-polymerizable cyclic compounds and cyclic imines such as ethyleneimine, cyclic lactonic acids such as  $\beta$ -propyolactone and glycolic acid lactide, or dimethylcyclopolsiloxanes may be used. As specific examples of combinations of two or more ion-polymerizable cyclic compounds, combinations of tetrahydrofuran and propylene oxide, tetrahydrofuran and 2-methyltetrahydrofuran, tetrahydrofuran and 3-methyltetrahydrofuran, tetrahydrofuran and ethylene oxide, propylene oxide and ethylene oxide, butene-1-oxide and ethylene oxide, a ternary copolymer of tetrahydrofuran, butene-1-oxide, and ethylene oxide, and the like can be given. The ring-opening copolymer of these ion-polymerizable cyclic compounds may be either a random copolymer or a block copolymer. Of these polyether diols, polypropylene glycol is preferable from the viewpoint of providing jelly resistance and water resistance to the cured product of the present invention. Polypropylene glycol with a polystyrene-reduced number average molecular weight determined by gel permeation chromatography (GPC) of 1000-7000 is particularly preferable.

As examples of commercially available products of these polyether diols, PTMG650, PTMG1000, PTMG2000 (manufactured by Mitsubishi Chemical Corp.), EXCENOL 1020, 2020, 3020, PREMINOL PML-4002, PML-5005 (manufactured by Asahi Glass Co., Ltd.), UNISAFE DC1100, DC1800, DCB1000 (manufactured by Nippon Oil and Fats Co., Ltd.), PPTG1000, PPTG2000, PPTG4000, PTG400, PTG650, PTG1000, PTG2000, PTG-L1000, PTG-L2000 (manufactured by Hodogaya Chemical Co., Ltd.), Z-3001-4, Z-3001-5, PBG2000 (manufactured by Daiichi Kogyo Seiyaku Co., Ltd.), ACCLAIM 2200, 2220, 3201, 3205, 4200, 4220, 8200, 12000 (manufactured by Lyondell), and the like can be given.

The above polyether diols are preferable as the polyol. In addition, polyester diols, polycarbonate diols, polycaprolactone diols, and the like may be used either individually or in combination with the polyether diols. There are no specific limitations to the manner of polymerization of these structural units, which may be any of random polymerization, block polymerization, or graft polymerization.

As examples of the polyisocyanate (B) used for synthesizing the urethane (meth)acrylate (1), aromatic diisocyanates, alicyclic diisocyanates, aliphatic diisocyanates, and the like can be given. There are no specific limitations to the polyisocyanate (B) insofar as the compound can be used in the resin composition for optical fibers. Of these 2,4-tolylene diisocyanate is preferred as aromatic diisocyanate but alicyclic diisocyanates are more preferred, and isophorone diisocyanate and hydrogenated bisphenol-F diisocyanate as being still more preferred. These

diisocyanate compounds may be used either individually or in combination of two or more.

As the hydroxyl group-containing (meth)acrylate (C) used for synthesizing the urethane (meth)acrylate (1), a hydroxyl group-containing (meth)acrylate in which the hydroxyl group is bonded to the primary carbon atom (hereinafter called "primary hydroxyl group-containing (meth)acrylate") and a hydroxyl group-containing (meth)acrylate in which the hydroxyl group is bonded to the secondary carbon atom (hereinafter called "secondary hydroxyl group-containing (meth)acrylate") are preferable in view of reactivity with an isocyanate group of the polyisocyanate.

As examples of the primary hydroxyl group-containing (meth)acrylate, 2-hydroxyethyl (meth)acrylate, 4-hydroxybutyl (meth)acrylate, 1,6-hexanediol mono(meth)acrylate, pentaerythritol tri(meth)acrylate, dipentaerythritol penta(meth)acrylate, neopentyl glycol mono(meth)acrylate, trimethylolpropane di(meth)acrylate, trimethylolethane di(meth)acrylate, and the like can be given.

As examples of the secondary hydroxyl group-containing (meth)acrylate, 2-hydroxypropyl (meth)acrylate, 2-hydroxybutyl (meth)acrylate, 2-hydroxy-3-phenyloxypropyl (meth)acrylate, 4-hydroxycyclohexyl (meth)acrylate, and the like can be given. Further examples include a compound obtained by the addition reaction of (meth)acrylic acid and a glycidyl group-containing compound such as alkyl glycidyl ether, allyl glycidyl ether, or glycidyl (meth)acrylate, and the like. These hydroxyl group-containing (meth)acrylate compounds may be used either individually or in combination of two or more.

The proportion of the polyol (A), polyisocyanate compound (B), and hydroxyl group-containing (meth)acrylate (C) used for synthesizing the urethane (meth)acrylate (1) is preferably determined so that an isocyanate group included in the polyisocyanate compound and a hydroxyl group included in the hydroxyl group-containing (meth)acrylate are respectively 1.1-2 equivalents and 0.1-1 equivalent for one equivalent of a hydroxyl group included in the polyol.

In addition, diamines may be used for synthesizing the urethane (meth)acrylate (1) in combination. As examples of diamines, diamines such as ethylenediamine, tetramethylenediamine, hexamethylenediamine, p-phenylenediamine, and 4,4'-diaminodiphenylmethane, diamines containing a hetero atom, polyether diamines, and the like can be given.

Part of the hydroxyl group-containing (meth)acrylate may be replaced

by a compound having a functional group which can be added to an isocyanate group. As examples of such a compound,  $\gamma$ -aminopropyltriethoxysilane,  $\gamma$ -mercaptopropyltrimethoxysilane, and the like can be given. Use of such a compound can further improves adhesion to substrates such as glass.

5 In the synthesis of the urethane (meth)acrylate (1), it is preferable to use a urethanization catalyst such as copper naphthenate, cobalt naphthenate, zinc naphthenate, dibutyltin dilaurate, triethylamine, 1,4-diazabicyclo[2.2.2]octane, or 2,6,7-trimethyl-1,4-diazabicyclo[2.2.2]octane in an amount of 0.01-1 wt% of the total amount of the reactants. The reaction temperature is usually 5-90°C, and preferably 10-80°C.

10 The polystyrene-reduced number average molecular weight of the urethane (meth)acrylate (1) determined by GPC is usually 500-40,000, and preferably 700-30,000 in order to ensure good breaking elongation of the cured product and appropriate viscosity of the curable resin composition of the present invention.

The content of the urethane (meth)acrylate (1) in the curable resin  
15 composition of the present invention is preferably 50-70 wt%, and particularly preferably 55-65 wt% in order to ensure excellent mechanical characteristics such as jelly resistance, water resistance, and breaking elongation of the cured product and appropriate viscosity of the curable resin composition of the present invention. If the content exceeds 70 wt%, since the cured product exhibits a Young's modulus of more  
20 than 2.0 MPa, the composition is unsuitable for a resin for coating optical fibers. Moreover, viscosity of the curable resin composition exceeds 6.0 Pa·s, thereby resulting in decreased workability. In addition, water resistance of the cured product decreases. If the content is less than 50 wt%, oil resistance and water resistance of the cured product decrease. The Young's modulus of the cured product used as a primary  
25 layer for optical fibers is preferably 0.1-2.0 MPa, and more preferably between 0.5-1.5 MPa. Viscosity of the curable resin composition is preferably 1.0-6.0 Pa·s.

As examples of the reactive diluent (2) copolymerizable with the component (1) used in the curable resin composition of the present invention, polymerizable monofunctional compounds or polymerizable monofunctional  
30 compounds to which polymerizable polyfunctional compounds are added can be given. As examples of monofunctional compounds, vinyl group-containing lactams such as N-vinylpyrrolidone and N-vinylcaprolactam, (meth)acrylates having an alicyclic structure such as isobornyl (meth)acrylate, bornyl (meth)acrylate, tricyclodecanyl (meth)acrylate, and dicyclopentanyl (meth)acrylate, benzyl (meth)acrylate, 4-butylcyclohexyl

(meth)acrylate, acryloylmorpholine, vinylimidazole, vinylpyridine, and the like can be given. Further examples include 2-hydroxyethyl (meth)acrylate, 2-hydroxypropyl (meth)acrylate, 2-hydroxybutyl (meth)acrylate, methyl (meth)acrylate, ethyl (meth)acrylate, propyl (meth)acrylate, isopropyl (meth)acrylate, butyl (meth)acrylate, 5 amyl (meth)acrylate, isobutyl (meth)acrylate, t-butyl (meth)acrylate, pentyl (meth)acrylate, isoamyl (meth)acrylate, hexyl (meth)acrylate, heptyl (meth)acrylate, octyl (meth)acrylate, isooctyl (meth)acrylate, 2-ethylhexyl (meth)acrylate, nonyl (meth)acrylate, decyl (meth)acrylate, isodecyl (meth)acrylate, undecyl (meth)acrylate, dodecyl (meth)acrylate, lauryl (meth)acrylate, stearyl (meth)acrylate, isostearyl (meth)acrylate, tetrahydrofurfuryl (meth)acrylate, butoxyethyl (meth)acrylate, 10 ethoxydiethylene glycol (meth)acrylate, benzyl (meth)acrylate, phenoxyethyl (meth)acrylate, polyethylene glycol mono(meth)acrylate, polypropylene glycol mono(meth)acrylate, methoxyethylene glycol (meth)acrylate, ethoxyethyl (meth)acrylate, methoxypolyethylene glycol (meth)acrylate, methoxypolypropylene glycol (meth)acrylate, diacetone (meth)acrylamide, isobutoxymethyl (meth)acrylamide, 15 N,N-dimethyl (meth)acrylamide, t-octyl (meth)acrylamide, dimethylaminoethyl (meth)acrylate, diethylaminoethyl (meth)acrylate, 7-amino-3,7-dimethyloctyl (meth)acrylate, N,N-diethyl (meth)acrylamide, N,N-dimethylaminopropyl (meth)acrylamide, hydroxybutyl vinyl ether, lauryl vinyl ether, cetyl vinyl ether, 2-ethylhexyl vinyl ether, and the like.

Of these monofunctional compounds, vinyl group-containing lactams such as N-vinylpyrrolidone and N-vinylcaprolactam, monofunctional (meth)acrylate containing an aliphatic hydrocarbon group having 10 or more carbon atoms are preferable. The aliphatic group having 10 or more carbon atoms may be linear, 25 branched, or alicyclic. The number of carbon atoms is preferably 10-24. Of these, isobornyl (meth)acrylate, isodecyl (meth)acrylate, and lauryl (meth)acrylate are still more preferable. Particularly preferable compounds are isobornyl (meth)acrylate and/or isodecyl (meth)acrylate. As examples of commercially available products of these monofunctional compounds, IBXA (manufactured by Osaka Organic Chemical Industry Co., Ltd.), Aronix M-111, M-113, M-114, M-117, and TO-1210 (manufactured by 30 Toagosei Co., Ltd.) can be given.

There are no specific limitations to polyfunctional compounds insofar as the compounds can be used in a resin composition for optical fibers. Preferable examples include polyethylene glycol diacrylate, tricyclodecanediyldimethylene 35 di(meth)acrylate, di(meth)acrylate of ethylene oxide addition bisphenol A, and tris(2-



hydroxyethyl)isocyanurate tri(meth)acrylate. As examples of commercially available products of these polyfunctional compounds, Light acrylate 9EG-A, 4EG-A (manufactured by Kyoisha Chemical Co., Ltd.), Yupimer UV, SA1002 (manufactured by Mitsubishi Chemical Corp.), and Aronix M-215, M-315, M-325 (manufactured by Toagosei Co., Ltd.) can be given.

The content of the reactive diluent in the curable resin composition of the present invention is preferably 1-50 wt%, and still more preferably 2-45 wt%. If the content is less than 1 wt%, curability may be impaired. If the content exceeds 50 wt%, application may become uneven due to low viscosity, thereby resulting in unstable application.

As examples of the polymerization initiator (3) used in the present invention, a heat polymerization initiator and a photopolymerization initiator can be given. As examples of heat polymerization initiator, peroxides, azo compounds, and like can be given. Specific examples include benzoyl peroxide, t-butyl oxybenzoate, azobisisobutyronitrile, and the like.

In the case of curing the curable resin composition of the present invention using light, a photosensitizer may be added as required in addition to the photopolymerization initiator. As examples of the photopolymerization initiator, 1-hydroxycyclohexyl phenyl ketone, 2,2-dimethoxy-2-phenylacetophenone, xanthone, fluorenone, benzaldehyde, fluorene, anthraquinone, triphenylamine, carbazole, 3-methylacetophenone, 4-chlorobenzophenone, 4,4'-dimethoxybenzophenone, 4,4'-diaminobenzophenone, Michler's ketone, benzoin propyl ether, benzoin ethyl ether, benzyl dimethyl ketal, 1-(4-isopropylphenyl)-2-hydroxy-2-methylpropan-1-one, 2-hydroxy-2-methyl-1-phenylpropan-1-one, thioxanethone, diethylthioxanthone, 2-isopropylthioxanthone, 2-chlorothioxanthone, 2-methyl-1-[4-(methylthio)phenyl]-2-morpholino-propan-1-one, 2,4,6-trimethylbenzoyldiphenylphosphine oxide, bis-(2,6-dimethoxybenzoyl)-2,4,4-trimethylpentylphosphine oxide, and the like can be given. As examples of commercially available products of the photopolymerization initiator, Irgacure 184, 369, 651, 500, 907, CGI1700, CGI1750, CGI1850, CG24-61, Darocur 1116, 1173 (manufactured by Ciba Specialty Chemicals Co., Ltd.), Lucirin TPO (manufactured by BASF), Ubecryl P36 (manufactured by UCB), and the like can be given. As examples of the photosensitizer, triethylamine, diethylamine, N-methyldiethanoleamine, ethanolamine, 4-dimethyl aminobenzoic acid, methyl 4-dimethylaminobenzoate, ethyl 4-dimethylaminobenzoate, isoamyl 4-dimethylaminobenzoate, and the like can be given. As examples of commercially

available products of the photosensitizer, Ubecryl P102, 103, 104, 105 (manufactured by UCB), and the like can be given.

The content of the polymerization initiator (3) in the curable resin composition of the present invention is preferably 0.3-8 wt%, and particularly preferably  
5 0.5-5 wt%.

Additives such as coloring agents, light stabilizers, silane coupling agents, heat polymerization inhibitors, leveling agents, surfactants, preservatives, plasticizers, lubricants, solvents, fillers, aging preventives, wettability improvers, and coating surface improvers may be added to the curable resin composition in addition to  
10 the above components. As examples of light stabilizers, Tinuvin 292, 144, 622LD (manufactured by Ciba Specialty Chemicals Co., Ltd.), Sanol LS770 (manufactured by Sankyo Co., Ltd.), TM-061 (manufactured by Sumitomo Chemical Industries Co., Ltd.), and the like can be given. As examples of silane coupling agents,  $\gamma$ -aminopropyltriethoxysilane,  $\gamma$ -mercaptopropyltrimethoxysilane,  $\gamma$ -methacryloxypropyltrimethoxysilane, commercially available products such as SH6062,  
15 6030 (manufactured by Toray-Dow Corning Silicone Co., Ltd.), KBE903, 603, 403 (manufactured by Shin-Etsu Chemical Co., Ltd.), and the like can be given.

The curable resin composition of the present invention is cured using heat and/or radiation. Radiation used herein includes infrared rays, visible rays,  
20 ultraviolet rays, X-rays, electron beams,  $\alpha$ -rays,  $\beta$ -rays,  $\gamma$ -rays, and the like. Of these, ultraviolet rays are particularly preferable.

A jelly resistance evaluation method of the present invention is described below.

A cured product used in the jelly resistance evaluation method of the present invention is a cured product of a sample curable resin composition for coating  
25 optical fibers. The thickness of the cured product is 200  $\mu$ m, which is close to the thickness when applied to optical fibers in practice. The length and the width of the cured product film are 25 mm and 6 mm, respectively.

In this evaluation method, mineral oil (class 1, No. 1) described in JIS  
30 C2320 is used as a standard substance for a jelly. More specifically, mineral oil manufactured by Aldrich or mineral oil manufactured by Wako Pure Chemicals Co., Ltd. can be used.

The jelly resistance of the cured product is evaluated by measuring the dimensional change of the cured product after immersion in mineral oil at 25°C for

one week. The rate of dimensional change is determined by measuring the dimensions of the cured product in the longitudinal direction and using the following equation.

- 5     Rate of dimensional change (%) = (dimension after immersion - dimension before immersion)/dimension before immersion x 100

10     The rate of dimensional change of the cured product after immersion in mineral oil at 25°C for one week is 2.5% or less, preferably 2.3% or less, and particularly preferably 2.0% or less. If the rate of dimensional change exceeds 2.5%, the jelly resistance of the cured product may decrease, whereby separation between the glass and the primary layer in the optical fiber occurs.

A water resistance evaluation method of the present invention is described below.

15     A cured product used in the water resistance evaluation method is a cured product of a sample curable resin composition for coating optical fibers. The cured product is used as a primary layer of a three-layer cured product consisting of secondary layer-primary layer-secondary layer (Figures 3 and 4), or a five-layer cured product consisting of coloring layer-secondary layer-primary layer-secondary layer-  
20     coloring layer (Figures 1 and 2). The thickness of each layer is close to the thickness when applied to optical fibers in practice. Specifically, the thickness of the coloring layer is 20  $\mu\text{m}$ , the thickness of the secondary layer is 100  $\mu\text{m}$ , and the thickness of the primary layer is 200  $\mu\text{m}$ . Water resistance of the cured product used as the primary layer for the optical fiber used in practice can be accurately evaluated by locating the  
25     cured product at the center of such multilayer cured film.

In the three-layer cured product and the five-layer cured product, standard products are used for the secondary layer and the coloring layer. A method of fabricating such multilayer cured products is illustrated below taking the five-layer cured product as an example. In the case of the three-layer cured product, it is unnecessary  
30     to form the coloring layer in the method illustrated below.

- 1) A composition for forming a coloring layer is placed on a glass substrate (5 cm x 4 cm). The glass substrate is spin-coated with the composition at 5000 rpm for 20 seconds. The composition is then cured by irradiation with ultraviolet rays at a dose of 0.5 J/cm<sup>2</sup> in a nitrogen atmosphere to produce a coloring layer with a thickness of

20  $\mu\text{m}$ .

- 2) A polyethylene terephthalate (PET) film (thickness: 100  $\mu\text{m}$ ) with a rectangular opening at the center of the coloring layer (2 cm x 3.5 cm) is placed on the coloring layer formed on the glass substrate. A composition for forming a secondary layer is applied to the opening. After removing unnecessary composition using a glass bar, the PET film is removed. The composition is cured by irradiation with ultraviolet rays at a dose of 0.5 J/cm<sup>2</sup> in a nitrogen atmosphere to produce a first secondary layer (2 cm x 3.5 cm) with a thickness of 100  $\mu\text{m}$ .
- 3) A PET film (thickness: 200  $\mu\text{m}$ ) with a rectangular opening (1 cm x 2 cm) is placed at the center of the first secondary layer. A composition for forming a primary layer is applied to the opening. After removing unnecessary composition using a glass bar, the PET film is removed. The composition is cured by irradiation with ultraviolet rays at a dose of 0.5 J/cm<sup>2</sup> in a nitrogen atmosphere to produce a primary layer (1 cm x 2 cm) with a thickness of 200  $\mu\text{m}$ .
- 4) A PET film (thickness: 400  $\mu\text{m}$ ) with a rectangular opening (2 cm x 3.5 cm) formed so as to enclose the primary layer obtained in the above 3) and to superpose the first secondary layer is placed on the coloring layer. A composition for forming a secondary layer is applied to the opening. After removing unnecessary composition using a glass bar, the PET film is removed. The composition is cured by irradiation with ultraviolet rays at a dose of 0.5 J/cm<sup>2</sup> in a nitrogen atmosphere to produce a second secondary layer (2 cm x 3.5 cm) with a thickness of 400  $\mu\text{m}$  at the circumference. The thickness of the second secondary layer at the center is about 200  $\mu\text{m}$ .
- 5) The glass substrate obtained in the above 4) is placed on a spin coater. 5 ml of the composition for forming a coloring layer is applied to the glass substrate. The glass substrate is spin-coated with the composition at 5000 rpm for 20 seconds. The composition is cured by irradiation with ultraviolet rays at a dose of 0.5 J/cm<sup>2</sup> in a nitrogen atmosphere. The thickness of this second coloring layer after curing is about 20  $\mu\text{m}$ .
- 6) Cured layers are cut at 1 cm from all four sides of the glass substrate using a cutter and removed from the glass substrate to obtain a test specimen consisting of five layers.

As the coloring layer used in the water resistance evaluation method of the present invention, BESTCURE FR CLEAR (manufactured by T & K TOKA

COMPANY) and the like can be used. As the secondary layer, a cured film with a Young's modulus of 100-1000 MPa can be used.

In this evaluation method, the multilayer cured product is immersed in hot water at 60°C for one week. The presence or absence of foam defects in the primary layer of the multilayer cured product is then determined. If there are no foam defects, the curable resin composition used for the primary layer is judged to exhibit good water resistance. The presence or absence of foam defects is determined using an optical microscope.

A cured product exhibiting no foam defects in the water resistance evaluation method of the present invention exhibits good water resistance. Therefore, when the cured product is used for coating optical fibers, problems such as an increase in the transmission loss due to separation of the cured product or formation of foams during immersion in water can be prevented.

#### Examples

The present invention is described below in more detail by examples, which are not intended to be limiting of the present invention. "Parts" and "%" respectively mean "parts by weight" and "wt%" unless otherwise indicated.

#### Synthesis Example 1

(synthesis example of polymerizable oligomer (a))

A reaction vessel equipped with a stirrer was charged with 831.0 g of polypropylene glycol with a number average molecular weight of 2000, 129.3 g of isophorone diisocyanate, 0.24 g of 2,6-di-*t*-butyl-*p*-cresol, and 0.08 g of phenothiazine.

The mixture was cooled to 15°C while stirring. After the addition of 0.8 g of dibutyltin dilaurate, the mixture was slowly heated to 35°C for one hour while stirring. The mixture was then allowed to react at 50°C. When the residual isocyanate concentration decreased to 1.26 wt% or less (ratio to raw material, hereinafter the same), 38.6 g of 2-hydroxyethyl acrylate was added. The mixture was allowed to react at about 60°C while stirring. The reaction was terminated when the residual isocyanate concentration was 0.1 wt% or less to obtain a polymerizable oligomer (hereinafter called "polymerizable oligomer (a)").

### Synthesis Example 2

(synthesis example of polymerizable oligomer (b))

A reaction vessel equipped with a stirrer was charged with 907.2 g of polypropylene glycol with a number average molecular weight of 4000, 70.6 g of isophorone diisocyanate, 0.24 g of 2,6-di-t-butyl-p-cresol, and 0.08 g of phenothiazine. The mixture was cooled to 15°C while stirring. After the addition of 0.8 g of dibutyltin dilaurate, the mixture was slowly heated to 35°C for one hour while stirring. Then, the mixture was allowed to react at 50°C. When the residual isocyanate concentration decreased to 0.6 wt% or less (ratio to raw material, hereinafter the same), 21.1 g of 2-hydroxyethyl acrylate was added. The mixture was allowed to react at about 60°C while stirring. The reaction was terminated when the residual isocyanate concentration was 0.1 wt% or less to obtain a polymerizable oligomer (hereinafter called "polymerizable oligomer (b)").

### Synthesis Example 3

(synthesis example of polymerizable oligomer (c))

A reaction vessel equipped with a stirrer was charged with 950.9 g of polypropylene glycol with a number average molecular weight of 8000, 37.0 g of isophorone diisocyanate, 0.24 g of 2,6-di-t-butyl-p-cresol, and 0.08 g of phenothiazine. The mixture was cooled to 15°C while stirring. After the addition of 0.8 g of dibutyltin dilaurate, the mixture was slowly heated to 35°C for one hour while stirring. The mixture was then allowed to react at 50°C. When the residual isocyanate concentration decreased to 0.4 wt% or less (ratio to raw material, hereinafter the same), 11.0 g of 2-hydroxyethyl acrylate was added. The mixture was allowed to react at about 60°C while stirring. The reaction was terminated when the residual isocyanate concentration was 0.1 wt% or less to obtain a polymerizable oligomer (hereinafter called "polymerizable oligomer (c)").

### Synthesis Example 4

(synthesis example of polymerizable oligomer (d))

A reaction vessel equipped with a stirrer was charged with 96.4 g of isophorone diisocyanate, 0.024 g of 2,6-di-t-butyl-p-cresol, 0.08 g of phenothiazine, and 0.8 g of dibutyltin dilaurate. The mixture was cooled to 15°C while stirring. 86.9 g of 2-hydroxyethyl acrylate was added using a dropping funnel for one hour. The mixture was slowly heated to 35°C for one hour while stirring. After the addition of 815.6g of a

copolymer of tetrahydrofuran and 2-methyltetrahydrofuran with a number average molecular weight of 2000 ("PTGL2000" manufactured by Hodogaya Chemical Co., Ltd.), the mixture was allowed to react at 60°C. The reaction was terminated when the residual isocyanate concentration was 0.1 wt% or less to obtain a polymerizable  
5 oligomer (hereinafter called "polymerizable oligomer (d)").

#### Examples 1-5 and Comparative Examples 1-3

A reaction vessel equipped with a stirrer was charged with compounds according to the composition (weight ratio) shown in Table 1. The mixture  
10 was stirred at 50°C to obtain compositions of Examples and Comparative Examples.

#### Reference Example 1

A synthesis example of a composition for secondary layer is described below.

15 A reaction vessel equipped with a stirrer was charged with 134.8 parts of 2,4-toluenediisocyanate, 0.8 part of dibutyltin dilaurate, and 0.32 part of 2,6-di-t-butyl-p-cresol. The mixture was cooled to 15°C or less. 89.9 parts of 2-hydroxyethyl acrylate was added dropwise at 30°C or less while stirring. After the addition, the mixture was allowed to react at 30°C for one hour. After the addition of 774.0 parts of a  
20 ring-opening polymer of tetrahydrofuran with a number average molecular weight of 2,000, the mixture was stirred at 20-55°C. The reaction was terminated when the residual isocyanate was 0.1 wt% or less. This liquid resin is called "U-1".

Then, a reaction vessel equipped with a stirrer was charged with 230.6 parts of IBXA (manufactured by Osaka Organic Chemical Industry Co., Ltd.),  
25 273.0 parts of 2,4-toluenediisocyanate, 0.6 part of dibutyltin dilaurate, and 0.24 part of 2,6-di-t-butyl-p-cresol. The mixture was cooled to 15°C or less. 182.0 parts of 2-hydroxyethyl acrylate was added dropwise at 30°C or less while stirring. After the addition, the mixture was allowed to react at 30°C for one hour. After the addition of 313.5 parts of Uniol DA400 (manufactured by Nippon Oil and Fats Co., Ltd.), the  
30 mixture was stirred at 20-55°C. The reaction was terminated when the residual isocyanate was 0.1 wt% or less. This liquid resin is called "U-2".

A reaction vessel equipped with a stirrer was charged with 499.7 parts of IBXA (manufactured by Osaka Organic Chemical Industry Co., Ltd.), 214.2 parts of 2,4-toluenediisocyanate, 0.4 part of dibutyltin dilaurate, and 0.16 part of 2,6-di-  
35 t-butyl-p-cresol. The mixture was cooled to 15°C or less. 285.6 parts of 2-hydroxyethyl

acrylate was added dropwise at 30°C or less while stirring. After the addition, the mixture was allowed to react at 30°C for one hour. The mixture was then stirred at 20-55°C. The reaction was terminated when the residual isocyanate was 0.1 wt% or less. This liquid resin is called "U-3".

5                   A reaction vessel equipped with a stirrer was charged with 32.48 parts of U-1, 28.15 parts of U-2, 9.84 parts of U-3, 7.87 parts of N-vinylpyrrolidone, 7.87 parts of tricyclodecanediyl dimethylene diacrylates, 10.83 parts of diacrylate of diol of ethylene oxide addition product of bisphenol A, and 0.3 parts of Irganox 1035 (manufactured by Ciba Specialty Chemicals Co., Ltd.). The mixture was stirred at 50-10   60°C until a homogeneous transparent liquid was obtained, to produce urethane acrylate. This liquid resin is called A-1. This liquid resin was applied to a glass plate using an applicator for a thickness of 381  $\mu\text{m}$  and irradiated with ultraviolet rays at a dose of 0.5  $\text{J}/\text{cm}^2$  in a nitrogen atmosphere using a 3.5 kW metal halide lamp ("SMX-3500/F-OS" manufactured by ORC Co., Ltd.) to obtain a cured film with a thickness of 15   about 200  $\mu\text{m}$ . The Young's modulus of this cured film measured in a tensile test was 800  $\text{MPa}/\text{mm}^2$ .

#### Viscosity measurement method of resin composition

20                   Viscosity of the resin composition was measured according to JIS K7117. Viscosity was measured using a DA viscometer and a DA No. 4 spindle at a spindle rotation rate of 50 rpm.

#### Measurement method of Young's modulus of cured product

25                   The liquid composition was applied to a glass plate using an applicator for a thickness of 381  $\mu\text{m}$ . The liquid composition was irradiated with ultraviolet rays at a dose of 0.1  $\text{J}/\text{cm}^2$  in air using a 3.5 kW metal halide lamp ("SMX-3500/F-OS" manufactured by ORC Co., Ltd.) to obtain a cured film with a thickness of about 200  $\mu\text{m}$ . The cured product was allowed to stand at a temperature of 23°C and a relative humidity of 50% for 12 hours or more to prepare a test specimen. The Young's 30   modulus of the test specimen at 23°C was measured according to JIS K7113. Note that the Young's modulus was calculated from a tensile stress at a tensile rate of 1 mm/min and a strain of 2.5%.



Test Example 1 (Test method for mineral oil immersion dimensional change)

The cured products of the compositions obtained in Examples and Comparative Examples were evaluated according to mineral oil immersion dimensional change. The liquid composition was applied to a glass plate using an applicator for a thickness of 381  $\mu\text{m}$ . The liquid composition was irradiated with ultraviolet rays at a dose of 0.1  $\text{J}/\text{cm}^2$  in air using a 3.5 kW metal halide lamp ("SMX-3500/F-OS" manufactured by ORC Co., Ltd.) to obtain a cured film with a thickness of about 200  $\mu\text{m}$ . The cured film was cut into a sheet with a length of 25 mm and a width of 6 mm. The dimensions of the cured film in the longitudinal direction was measured before mineral oil immersion. The cured film was then immersed in mineral oil (manufactured by Aldrich) for one week. The immersion temperature was 25°C. The dimensions of the cured product in the longitudinal direction were measured to calculate the rate of dimensional change according to the above equation.

Test Example 2 (hot water immersion test of multilayer film)

A five-layer film shown in Figure 2 was fabricated as follows. A coloring layer ("BESTCURE FR CLEAR" manufactured by T & K TOKA COMPANY) was applied as a first coloring layer to a glass substrate to a thickness of 20  $\mu\text{m}$  using a spin coating method. The coloring layer was irradiated with ultraviolet rays at a dose of 0.5  $\text{J}/\text{cm}^2$  in a nitrogen atmosphere using a 3.5 kW metal halide lamp ("SMX-3500/F-OS" manufactured by ORC Co., Ltd.) to obtain a first coloring layer. The composition for forming a secondary layer obtained in Reference Example 1 was applied to the coloring layer as the first secondary layer, to a thickness of 100  $\mu\text{m}$  using a mask. The composition was irradiated with ultraviolet rays under the same conditions as the coloring layer. The composition obtained in Examples or Comparative Examples was applied to the secondary layer as the primary layer, to a thickness of 200  $\mu\text{m}$  using a mask. The composition was irradiated with ultraviolet rays under the same conditions as the coloring layer. A second secondary layer was formed on the primary layer under the same conditions as the first secondary layer. The second coloring layer was then formed under the same conditions as the first coloring layer to prepare a five-layer film.

The five-layer film was immersed in hot water at 60°C for one week. The presence or absence of foam defects was then observed using an optical microscope.

(Jelly resistance of optical fiber)

A quartz bar was heated to 2000°C and drawn at a drawing rate of 60 m/min using an optical fiber drawing machine to produce a quartz fiber with an outer diameter of 125  $\mu\text{m}$ . The composition was applied to the quartz fiber as the primary layer immediately after production. The composition was cured by irradiation with ultraviolet rays from a ultraviolet lamp (output: 3 Kw). The composition obtained in Reference Example 2 was applied as the secondary layer and cured by irradiation with ultraviolet rays in the same manner as in the above.

An optical fiber (outer diameter of primary layer: 200  $\mu\text{m}$ , outer diameter of secondary layer: 250  $\mu\text{m}$ ) was thus obtained. The optical fiber thus obtained was cut into a piece with a length of 1 m and immersed in a jelly buffer agent ("415DPL" manufactured by Syncofax). Note that the sections were not immersed in the buffer agent. The optical fiber was allowed to stand at 80°C for one month. The optical fiber was then removed from the buffer agent and observed using an optical microscope (magnification: 200) to determined the presence or absence of separation between the fiber and the primary layer.

(Hot water resistance of optical fiber)

The optical fiber obtained in the above was cut into a piece with a length of 1 m and immersed in water. Note that the sections were not immersed in water. The optical fiber was allowed to stand at 60°C for one month. The optical fiber was removed from water and observed using an optical microscope (magnification: 200) to determine the presence or absence of foam defects in the primary layer.

The results are shown in Table 1.

[Table 1]

Component		Example					Comparative Example		
		1	2	3	4	5	1	2	3
Urethane acrylate	Polymerizable oligomer (a)	61.8		61.8		59.0	82.0	40.0	
	Polymerizable oligomer (b)		61.8						
	Polymerizable oligomer (c)				61.8				
	Polymerizable oligomer (d)								49.3
Reactive diluent	N-vinylcaprolactam	7.4	7.5	7.4	7.1	9.2	6.9	7.0	7.9
	IBXA	14.4	13.4		18.3	9.9	4.9	35.5	19.8
	Isodecyl acrylate			14.4					
	M113	14.0	13.9	14.0	10.7	20.0	3.7	15.0	17.9
Polymerization initiator	1,6-Hexanediol diacrylate		1.0						2.0
	LUCIRIN TPO	1.3	1.3	1.3	1.0	0.8	1.3	1.3	2.0
	Diethylamine	0.1	0.1	0.1	0.1	0.1	0.2	0.2	0.1
	Silane coupling agent	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0
Viscosity of resin composition (PaDs)		4.0	4.5	3.5	5.0	3.5	10.0	2.5	3.5
	Young's modulus of cured product (MPa)	1.3	1.2	1.2	1.0	1.0	2.1	1.0	1.0
	Mineral oil immersion dimensional change (%)	1.5	2.2	2.1	2.4	2.3	2.0	2.8	2.9
	Jelly resistance of optical fiber (separation)	None	None	None	None	None	Observed	Observed	Observed
Hot water immersion foam defect		None	None	None	None	None	Defects	Defects	Defects
	Hot water resistance of optical fiber	None	None	None	None	None	Defects	Defects	Defects

Lucirin TPO: 2,4,6-Trimethylbenzoyldiphenylphosphine oxide (manufactured by BASF)

Lucirin TPO: 2,4,6-Trimethylbenzoyldiphenylphosphine oxide (manufactured by BASF)

IBXA: Isobornyl acrylate (manufactured by Osaka Organic Chemical Industry Co., Ltd.)

M113 : Nonylphenyl EO-modified acrylate (manufactured by Toagosei Co., Ltd.)

5 SH6062:  $\gamma$ -mercaptoptrimethoxysilane (manufactured by Toray-Dow Corning Silicone Co., Ltd.)

10 As is clear from Table 1, since the mineral oil immersion dimensional change and water resistance evaluation methods using a test specimen of the cured product of the present invention accurately reflect jelly resistance and water resistance of optical fibers used in practice, the cured product of the curable resin composition of the present invention excels in jelly resistance and water resistance.

CLAIMS

1. A curable resin composition for coating optical fibers comprising:

(1) 50-70 wt% of a urethane (meth)acrylate obtained by reacting (A) a polyol  
compound, (B) a polyisocyanate compound, and (C) a (meth)acrylate  
compound containing a hydroxyl group;

(2) a reactive diluent copolymerizable with the component (1); and

(3) a polymerization initiator;

wherein the rate of dimensional change of the cured product of the  
composition after immersion in mineral oil at 25°C for one week is 2.5% or  
less.

2. A curable resin composition for coating optical fibers comprising:

(1) 50-70 wt% of a urethane (meth)acrylate obtained by reacting (A) a polyol  
compound, (B) a polyisocyanate compound, and (C) a (meth)acrylate  
compound containing a hydroxyl group;

(2) a reactive diluent copolymerizable with the component (1); and

(3) a polymerization initiator;

wherein, when forming a product with a three-layer structure, specifically,  
secondary layer-primary layer-secondary layer, or a five-layer structure,  
specifically, coloring layer-secondary layer-primary layer-secondary layer-  
coloring layer, using the cured product of the composition as the primary  
layer, the primary layer exhibits no foam defects after immersing the  
product in hot water at 60°C for one week.

3. The curable resin composition for coating optical fibers according to claim 1 or  
2, comprising a monofunctional (meth)acrylate containing an aliphatic  
hydrocarbon group having 10 or more carbon atoms as the component (2).

4. The curable resin composition for coating optical fibers according to any one  
of claims 1 to 3, comprising 10-25 wt% of isobornyl (meth)acrylate and/or  
isodecyl (meth)acrylate as the component (2).

5. The curable resin composition for coating optical fibers according to any one  
of claims 1 to 4, wherein the polyol compound (A) of the component (1) is  
polypropylene glycol.

6. The curable resin composition for coating optical fibers according to claim 5,  
wherein the number average molecular weight of the polypropylene glycol is  
1,000-7,000.

7. The curable resin composition according to any one of claims 1-6, wherein the composition when cured has a Young's modulus of between 0.1-2.0 MPa.
8. A coated optical glass fiber having as a first coating, a cured product of the curable resin composition for coating optical fibers according to any one of claims 1 to 7, and a cured secondary coating.
9. A coated optical glass fiber having as a first coating a cured product of a curable resin composition for coating optical fibers comprising
- (1) 50-70 wt% of a urethane (meth)acrylate obtained by reacting (A) a polyol compound, (B) a polyisocyanate compound, and (C) a (meth)acrylate compound containing a hydroxyl group;
- (2) a reactive diluent copolymerizable with the component (1); and
- (3) a polymerization initiator;
- wherein the coated optical fibers shows no defects like separations of the coating from the fiber, or foam (bubble) formation in the coating after
- immersion in a jelly buffer agent at 80°C for one month or
- immersion in water at 60°C for one month.
10. Ribbon, loose tube cable, or ribbon cable comprising at least one coated optical glass fiber according to claim 8 or 9.

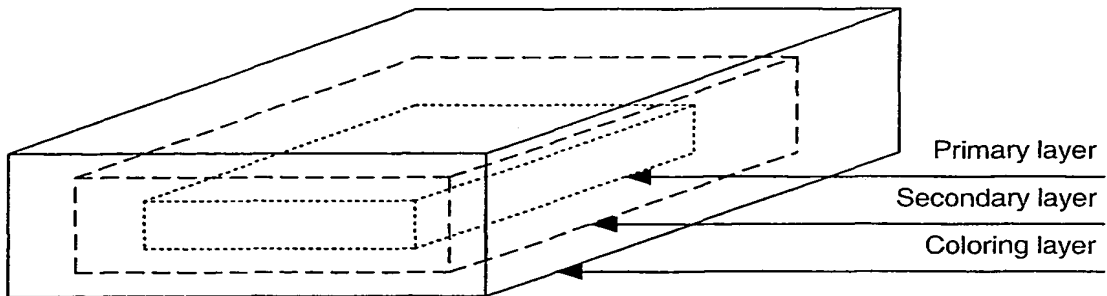


Figure 1 - 4

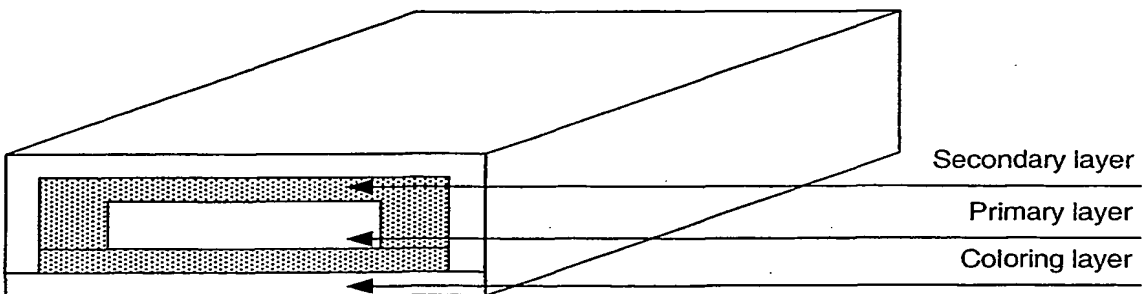


Figure 2 - 4

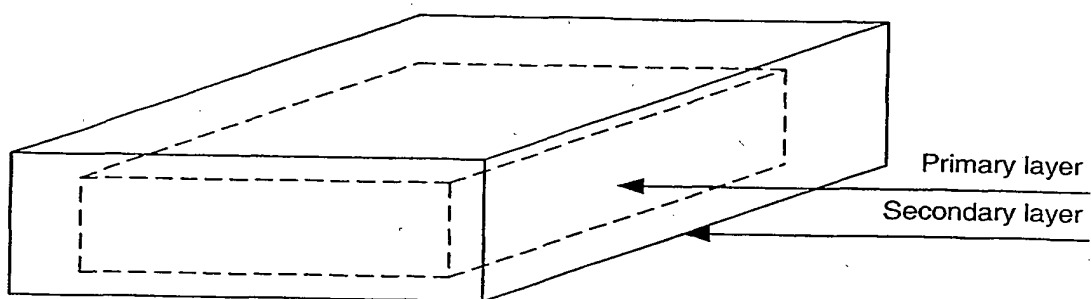


Figure 3 - 4

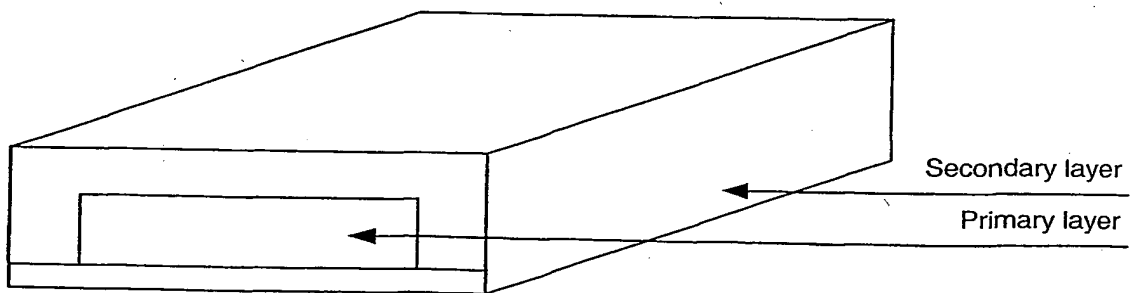


Figure 4 - 4



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(54) Title: CURABLE RESIN COMPOSITION FOR COATING OPTICAL FIBERS AND CURED PRODUCT THEREOF

(57) Abstract: The invention relates to a curable resin composition for coating optical fibers comprising: (1) 50-70 wt.% of a urethane (meth)acrylate obtained by reacting (A) a polyol compound, (B) a polyisocyanate compound, and (C) a (meth)acrylate compound containing a hydroxyl group; (2) a reactive diluent copolymerizable with the component (1); and (3) a polymerization initiator; wherein the rate of dimensional change of the cured product of the composition after immersion in mineral oil at 25 °C for one week is 2.5 % or less, or when forming a product with a three-layer structure, specifically, secondary layer-primary layer-secondary layer, or a five-layer structure, specifically, coloring layer-secondary layer-primary layer-secondary layer-coloring layer, using the cured product of the composition as the primary layer, the primary layer exhibits no foam defects after immersing the product in hot water at 60 °C for one week.

## INTERNATIONAL SEARCH REPORT

International Application No

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## A. CLASSIFICATION OF SUBJECT MATTER

IPC 7 C03C25/10 C08G18/67 C08G18/81

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 7 C03C C08G

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

WPI Data, EPO-Internal, PAJ

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	WO 97 16469 A (DSM) 9 May 1997 (1997-05-09) page 7, line 1 -page 10, line 14; claims 1-8,10-12; example 12; table 4	1,3,4
A	US 4 690 501 A (ZIMMERMAN ET AL) 1 September 1987 (1987-09-01) column 2, line 22 -column 4, line 9 column 5, line 18 - line 63; examples 1,2	1,3,4
A	US 5 787 218 A (OHTAKA ET AL) 28 July 1998 (1998-07-28) column 2, line 25 -column 5, line 60; examples 1,2	1,3,4

☐ Further documents are listed in the continuation of box C.☒ Patent family members are listed in annex.

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Date of the actual completion of the international search

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INTERNATIONAL SEARCH REPORT  
Information on patent family members

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**United States Patent** [19]  
**Rosebrooks**

[11] Patent Number: **4,510,884**  
[45] Date of Patent: **Apr. 16, 1985**

- [54] **DEVICE FOR PROVIDING A DUAL COATING ON AN OPTICAL FIBER**  
[75] Inventor: **Nathan B. Rosebrooks, West Thompson, Conn.**  
[73] Assignee: **ITT Corporation, New York, N.Y.**  
[21] Appl. No.: **485,700**  
[22] Filed: **Apr. 18, 1983**  
[51] Int. Cl.<sup>3</sup> ..... **B05C 3/172**  
[52] U.S. Cl. .... **118/405; 118/420**  
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[57] **ABSTRACT**

A device for coating optical fibers with at least two coatings. The device comprises a holding block and at least three coating dies. The holding block has an outer surface with first and second ends, and an inner surface defining a channel extending from the first end to the second end. The coating dies are press fit into the channel, separated by resin chambers. The holding block is further provided with passages providing fluid communication between the resin chambers and the outer surfaces of the holding block.

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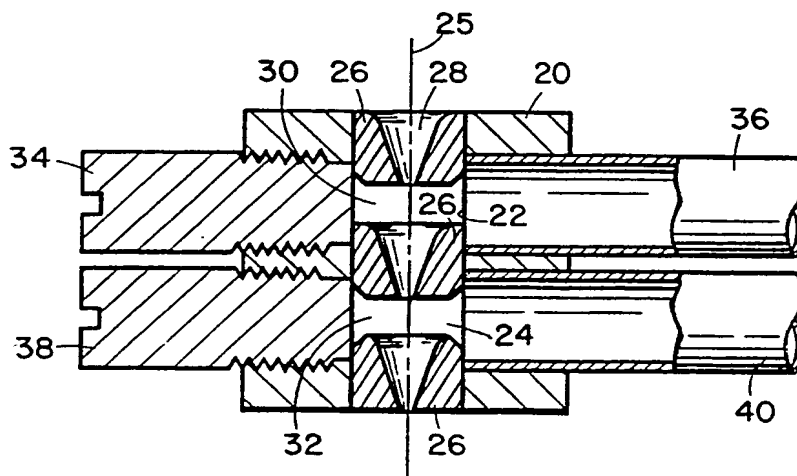
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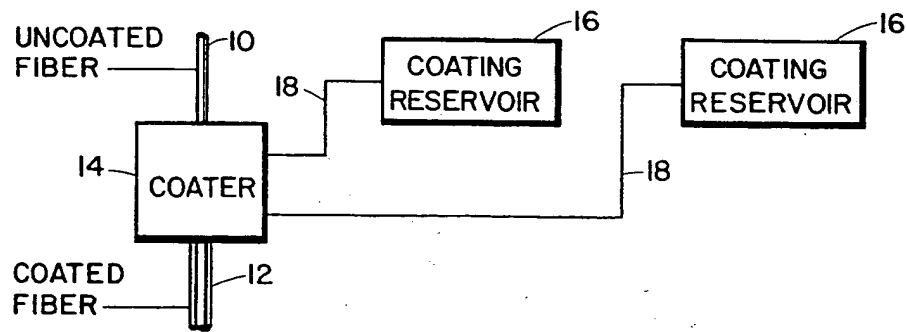
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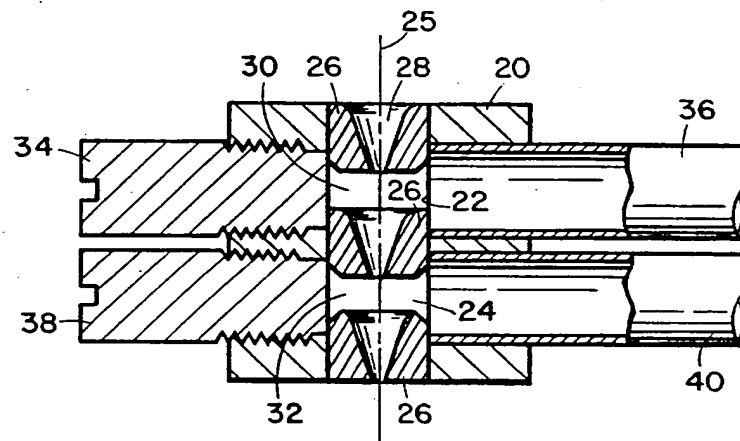
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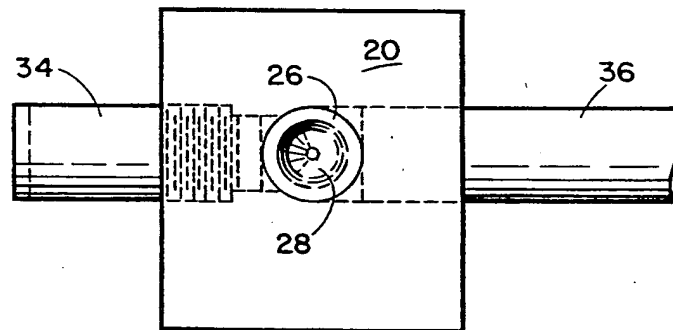




**Fig. 1**



**Fig. 2**



**Fig. 3**

# DEVICE FOR PROVIDING A DUAL COATING ON AN OPTICAL FIBER

## BACKGROUND OF THE INVENTION

The invention relates to devices for providing protective coatings on glass optical waveguide fibers.

Typically, optical waveguide fibers are produced by first manufacturing a glass preform and then drawing the preform into an optical fiber. The fiber can be drawn by placing an end of the preform into a hot furnace to soften the end thereof. The preform is then slowly fed into the furnace as an optical fiber is drawn out of the furnace from the softened end.

Optical waveguide fibers drawn from glass preforms in the manner described above are generally quite strong. For subsequent handling of the fibers, for example in producing optical fiber cables, it is necessary to maintain this high strength. However, subsequent handling can produce surface defects which significantly reduce the strength of the fiber. In order to reduce or eliminate surface defects during subsequent handling, one or more protective coatings are typically applied to the fibers immediately after they are drawn, and before they are wound on spools.

A protective coating may be provided on an optical fiber by a coating applicator device. The coating applicator may comprise, for example, a pair of wire-coating dies mounted in line in a holding block. A coating material is supplied under pressure between the two dies. As the fiber passes through the coating material, a coating is applied. Excess coating material is sheared off by the exit coating die.

While the optical fiber is provided with a protective coating after it is drawn from the hot furnace and before it is wound on a spool, the protective coating cannot be applied at any arbitrarily chosen time between these two production steps. As the fiber is drawn from the hot furnace, it must be allowed to cool for a predetermined amount of time before the coating or coatings are applied. If the fiber temperature is too high when the coating is applied, the coating material can be damaged by the excess heat.

In order to allow the hot fiber to cool before it is coated, the coating applicator must be fixed at a distance,  $d$ , from the furnace. The distance,  $d$ , is a function of the necessary cooling time,  $t$ , and the drawing velocity,  $v$ , of the fiber being drawn. This distance is given by  $d=vt$ . Thus, increasing the drawing velocity or the necessary cooling time increases the required distance between the furnace and the coating applicator. Since the fiber is drawn vertically down out of the furnace in a structure called a draw tower, increasing the draw velocity or the cooling time calls for increasing the height of the draw tower.

If two or more coatings are to be applied to an optical fiber, each coating is applied by a separate coating applicator, and is then cured before the next coating is applied. When two or more coating applicators are used, they are thus separated by a distance sufficient to permit curing of the previous coating before the application of the subsequent coating. The height of the draw tower, in such instances must then be increased by the additional curing distance plus the length of the second coating device. Alternatively, the draw velocity can be reduced to allow the first coating device to be placed closer to the furnace by this distance, while still

allowing adequate time for the fiber to cool before the first coating is applied.

## SUMMARY OF THE INVENTION

It is an object of the invention to provide a device for applying two or more coatings on an optical fiber, which device can be used without increasing the height of the draw tower or decreasing the draw velocity as compared to applying a single coating on the fiber.

It is a further object of the invention to provide a device for applying two or more separate uncured coatings on an optical fiber, which coatings are subsequently cured together after both are applied.

It is another object of the invention to provide a device for applying an uncured coating over another uncured coating without mixing the two coatings.

According to the invention, a device for coating optical fibers with at least two coatings comprises a holding block and at least three coating dies. The holding block has an outer surface with first and second ends, and an inner surface defining a channel extending from the first end to the second end. The coating dies are arranged in the channel in the holding block and are separated in the channel by first and second resin chambers. The holding block is further provided with passages providing fluid communication between each resin chamber and the outer surface of the holding block. Through these passages, coating materials can be supplied to the resin chambers.

Preferably, each coating die has a funnel-shaped passage therethrough. Each coating die is mounted in the channel in the holding block axially spaced along the channel axis and with their passages arranged coaxially. The wide openings of the funnel-shaped passages are disposed toward one end of the holding block and the narrow openings are disposed toward the other end of the holding block.

It is also preferred that the coating device according to the invention further include two pressurized coating reservoirs, and means for providing fluid communication between each reservoir and a passage in the holding block leading to a resin chamber.

In a preferred embodiment of the invention, the channel in the holding block is circularly cylindrical. In another embodiment, the pressure in the reservoir supplying the resin chamber nearer the exit of the coating device is higher than the pressure in the reservoir supplying the resin chamber nearer the input to the coating device.

The invention is advantageous because it provides two distinct coatings on an optical fiber in a device only insignificantly longer than a single-coating device. Two uncured coatings are applied, one over the other, and are subsequently cured simultaneously. As a result, an extra curing distance is avoided so the draw tower need not be raised and the draw velocity need not be reduced.

## BRIEF DESCRIPTION OF THE DRAWING

FIG. 1 is a schematic diagram of a dual coating device according to the invention.

FIG. 2 is a cross-sectional view of a dual coating device according to the invention.

FIG. 3 is a top plan view of a dual coating device according to the invention.

### DESCRIPTION OF THE PREFERRED EMBODIMENTS

FIG. 1 is a schematic diagram of a dual coating device according to the invention shown in the process of providing a coating on an optical fiber. In this Figure, an uncoated fiber 10 is passed through the coating device to produce a coated fiber 12 which emerges from the coating device. The coating device includes the coater 14, with or without the two coating reservoirs 16. Coating reservoirs 16 are, for example, pressurized vessels such as paint tanks containing a coating material, further described below. The coating reservoirs 16 are connected to the coater 14 by means 18 for providing fluid communication, for example high-pressure tubing.

FIG. 2 shows the coater 14 in more detail. Coater 14 includes a holding block 20 having an outer surface with first and second ends. In FIG. 2, the first end is the top of the holding block 20 and the second end is the bottom of the holding block 20.

Holding block 20 also has an inner surface 22 which defines a channel 24 extending from the first end of the holding block to the second end. Channel 24 has an axis 25, and may be simply formed by drilling the holding block 20. Holding block 20 is preferably made from stainless steel for reasons of chemical durability. Other materials, however, such as aluminum, brass, or plastic, could be used as well.

Coater 14 also includes at least three coating dies 26. These dies 26 are, for example, press fit into channel 24 in holding block 20. To ensure a tight fit, the diameter of the channel should be slightly smaller than the diameter of each coating die 26.

Each coating die 26 has first and second ends and a funnel-shaped passage 28 connecting the ends. The funnel-shaped passage 28 has a wide opening at the first end and narrows to a small opening (a sizing orifice) at the second end. Each coating die 26 is press fit into the channel 24 with their first ends disposed toward one end of the holding block. In FIG. 2, the first, or wide-opening ends of each coating die 26 are all disposed toward the top of the holding block 20, which is the input end of the coater 14. Moreover, the coating dies 26 are arranged in the holding block 20 with their funnel-shaped passages coaxial, so an optical fiber can be drawn straight through the coating device.

Coating dies 26 are, preferably, wire-drawing dies which are available from a number of suppliers throughout the country. These dies are used because they are inexpensive, readily available in many sizes, and durable. They are typically formed from tungsten carbide, but other materials may also be used.

As shown in FIG. 2, coating dies 26 are spaced apart in channel 24. The spaces between the coating dies 26 are resin chambers 30 and 32. The second end of each of the first two coating dies 26 (the top and middle dies 26 in FIG. 2) is adjacent to a resin chamber 30 or 32. These second ends are substantially perpendicular to the channel axis 25.

Resin or some other coating material is supplied to resin chambers 30 and 32 by means of passages provided in holding block 20. These passages are formed, for example, by cross-drilling holding block 20. In FIGS. 2 and 3, a plug 34 and a tube 36 are provided in the uppermost passage which communicates with resin chamber 30. Plug 34 seals one end of the passage, while the other end of the passage provides fluid communication between resin chamber 30 and tube 36 which is mounted

in the passage. Plug 38 and tube 40, mounted in the lower passage, provide the same functions as plug 34 and tube 36, respectively, for chamber 32.

The tubes 36 and 40 are connected to coating reservoirs 16 (not shown in FIGS. 2 or 3). Plugs 34 and 38, and tubes 36 and 40, are typically made of stainless steel, although other materials can also be used.

In order to use the dual-coating device according to the invention, the values of at least six parameters must be selected. These six parameters are (1) the coating material, (2) the viscosity of the coating material, (3) the application pressure of the coating material, (4) the velocity at which the optical fiber to be coated is being drawn, (5) the outer diameter of the fiber, and (6) the diameters of the sizing orifices (that is, the diameters of the narrow ends of the funnel-shaped passages in the coating dies 26). Moreover, each of these parameters (except for the fiber drawing speed) need not be the same for both the portion of the coating device providing the first coating and the portion of the coating device providing the second coating.

While there do not appear to be any inherent limits on the values of the parameters, there are certain considerations which should govern the selection of these values. First, it has been found that providing a higher pressure in resin chamber 32 (the chamber providing the second coating) than in resin chamber 30 (providing the first coating) often assures minimal mixing between the uncured materials of the two coating layers. Mixing is undesirable because each coating layer generally performs a different function and hence the materials are designed to have different properties. For example, typically the inner coating cures into a relatively soft, cushioning layer, which is often sticky and has low abrasion-resistance. The outer coating cures into a hard, protective layer, which is smooth and easy to handle.

On the other hand, if the pressure in chamber 30 greatly exceeds that in chamber 32 so that liquid coating material from chamber 32 is forced into chamber 30, it has been found that an unstable condition can arise in which the fiber may wander in the funnel-shaped passage of the primary coating die 26 (the middle die 26 in FIG. 2) and may rub against the die causing damage to the fiber. This instability can also cause undesirable mixing of the two coating materials. Moreover, if the pressure in chamber 30 is so great as to force coating material out of chamber 30 through the entrance (top) die 26, a similar instability may result with possible abrasion to the fiber.

Thus, there is always a range of acceptable pressures in the resin chambers which produce good coatings. The minimum of the range must be exceeded to assure an adequate supply of coating material, and the maximum must not be reached to avoid back flowing of the coating material.

Coating materials which can be used with the dual coating device according to the invention include, but are not limited to, ultraviolet-curable and heat-curable resins. For example, ultraviolet-curable acrylates, silicons, and epoxys may be used as coating materials. Solvent-borne coating materials, such as lacquers, may also be used. Regardless of the coating materials chosen, the viscosities of the materials should be matched close enough to minimize mixing between the uncured coatings.

The remaining parameters, namely the diameters of the sizing orifices, the fiber size, and the drawing speed are all selected based upon (1) the distance available



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between the furnace and the dual-coating device (in order to allow the fiber to cool before entering the coating device) and (2) the desired coating thickness. The orifice diameter and draw speed determine how large the flow of coating material must be. If these parameters are too large, the system may not be able to supply sufficient coating material, and the system will become unstable.

While the embodiment of the invention which has been described is a dual-coating device, it is contemplated that the principle of the invention could be extended to provide three or more coating layers on an optical fiber by providing a longer holding block 20, additional coating dies 26, and additional resin chambers.

#### EXAMPLE 1

A dual-coating device according to the invention was produced having a primary sizing orifice diameter of 240 microns and having a secondary sizing orifice diameter of 292 microns. (The primary sizing orifice is the small end of the funnel-shaped passage in the middle die 26 in FIG. 2. The secondary orifice is the small end of the passage in the bottom die 26.) A glass optical waveguide fiber having an outer diameter of 125 microns was drawn at 0.5 meters per second through the coating device. The coating materials were ultraviolet-curable urethane-acrylates applied at pressures of 3.75 pounds per square inch (measured 10 inches upstream from the entrance to the coater). The primary coating was DeSoto Desolite TM 950×030 (having a viscosity of 9000 cps±1500 cps), and the secondary coating was Desolite TM 950×042 (having a viscosity of 10,000 cps±1500 cps).

As a result of drawing the fiber through the coating device, an optical fiber having a primary coating of 190 microns and having a secondary coating of 223 microns was produced. The overall coating concentricity (herein defined as the thickness of the coating at its thinnest portion divided by the thickness of the coating at its thickest portion) was 0.77.

#### EXAMPLE 2

Using the same coating device and coating materials a 125 micron fiber was drawn through the coating device at 2 meters per second. The resulting coated fiber had a primary coating of 187 microns and a secondary coating of 209 microns. The overall concentricity was 0.88.

#### EXAMPLES 3 and 4

In Examples 3 and 4, a dual coating device according to the invention was produced having a primary sizing orifice diameter of 250 microns and having a secondary sizing orifice diameter of 350 microns. The primary coating was Desolite TM 950×030, and the secondary coating was a urethane-acrylate having a viscosity of 9000 cps±1500 cps. The coatings were applied at pressures of 3.75 pounds per square inch (measured as in Example 1).

In Example 3, fibers having outer diameters of from 54 microns to 248 microns were pulled through the dual coating device at 1.5 meters per second. Fibers having diameters from 83 to 181 microns were coated with no problems. Overall concentricities of 0.94 and greater were achieved. Smaller diameter fibers, however, were difficult to stabilize for any length of time. Larger diameter fibers experienced breaking and little, if any, pri-

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mary coating (although they had good secondary coatings).

In Example 4, fibers having outer diameters of approximately 130 microns were drawn through the dual coating device at velocities from 0.2 to 2.0 meters per second. Good results, including overall coating concentricities above 0.83, were obtained. It was observed, however, that at slow draw velocities the coating material began to cure within the coating device, which should be avoided.

In the Examples, the degree of mixing of the primary and secondary coatings was observed by injecting a colored dye into the primary coating material in the coating reservoir. A transparent plexiglass dual coating device was constructed to observe the flow of the liquid coating materials. No mixing between the primary and secondary coatings was observed. After curing the coatings, the coated fiber was observed under a microscope. A sharp boundary was observed between the primary and secondary coatings, indicating that no substantial mixing occurred.

I claim:

1. A device for coating optical fibers with at least two coatings, said device comprising:

a holding block having an outer surface with first and second ends, and having an inner surface defining a channel extending from the first end to the second end, said channel having an axis extending from the first end to the second end; and

at least first, second, and third coating dies arranged in the channel in the holding block, said first and second coating dies being axially separated in the channel by a first resin chamber, said second and third coating dies being axially separated in the channel by a second resin chamber;

characterized in that:

the holding block is further provided with a first passage providing fluid communication between the first resin chamber and the outer surface of the holding block, and a second passage providing fluid communication between the second resin chamber and the outer surface of the holding block;

each coating die has a first end and a second end; each coating die is press fit into the channel in the holding block with its first end disposed toward the first end of the holding block; and

the second end of each coating die which is adjacent to a resin chamber has a substantially planar end face and is substantially perpendicular to the channel axis.

2. A device for coating optical fibers as claimed in claim 1, characterized in that each coating die has a funnel-shaped passage connecting its first and second ends, the funnel-shaped passage having a wide opening at the first end and narrowing to a smaller opening at the second end.

3. A device as claimed in claim 2, characterized in that the device further comprises:

first and second pressurized coating reservoirs; first means for providing fluid communication between the first reservoir and the first passage; and second means for providing fluid communication between the second reservoir and the second passage.

4. A device as claimed in claim 3, characterized in that the channel is circularly cylindrical.

5. A device as claimed in claim 4, characterized in that:  
the second reservoir is nearer to the second end of the holding block than the first reservoir; and  
the pressure in the second reservoir is greater than the pressure in the first reservoir.

6. A device as claimed in claim 5, characterized in

that the funnel-shaped passages in the coating dies have axes, and the coating dies are arranged in the channel in the holding block with the axes of the passages lying on a straight line.

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⑤ A double-coating cup.

⑤ The present invention relates to a double-coating cup for coating an optical fibre with two protective layers simultaneously. The cup includes a body provided with an axially extending, frusto-conical boring whose largest diameter is located on the fibre-inlet side, a die holder firmly pressed into the body and having a frusto-conical outer surface which corresponds to the boring, and two die nozzles inserted in the die holder and functioning to apply the two layers of coating material. According to the invention, the body (1) has two first diametrically opposed first coating-material inlet channels (4) spaced at a first axial distance from the inlet side, and two second diametrically opposed second coating-material inlet channels (5) spaced at a second axial distance from the inlet side. The die holder (8) has a frusto-conical boring (10) whose largest diameter lies on the fibre inlet-side, a first cylindrical boring (11) which is a continuation of the conical boring, a first shoulder (12), a second cylindrical boring (13), a second shoulder (14), and a third cylindrical boring (15), wherein the outer surface (9) of the die holder has first ring-shaped slots (17) and first radial borings (18) which connect with the ring-shaped slots (17) and which open into the junction between the frusto-conical boring (10) and the first cylindrical boring (11), and second ring-shaped slots (19) which connect with second radial borings (20) which open into the second cylindrical boring (13). A guide die (21) is inserted into the conical boring (10) of the die holder and includes an outwardly extending part (25) in the end-wall (26) of the die. A first cylindrical die nozzle (27) having a frusto-conical recess (28) is inserted in the first cylindrical boring (11) in a manner such as to form a first coating-material gap or slot (30) between the die nozzle (27) and the guide die (21). A cylindrical intermediate guide die (34) is inserted against the first shoulder (12) and has a frusto-conical outwardly projecting part (37) in the end-wall (38) of the die. A second cylindrical die nozzle (39) having a frusto-conical recess (40) is positioned in the second cylindrical boring (13) and in abutment with the second shoulder (14) in a manner such as to form a second coating-material gap or slot (43) between the die nozzle (39) and the intermediate guide die (34). The die nozzle has distribution channels (44) which connect with the second radial borings (20) and which distribute coating material around the fibre coated with the first coating material and therewith centre the fibre in respective dies.

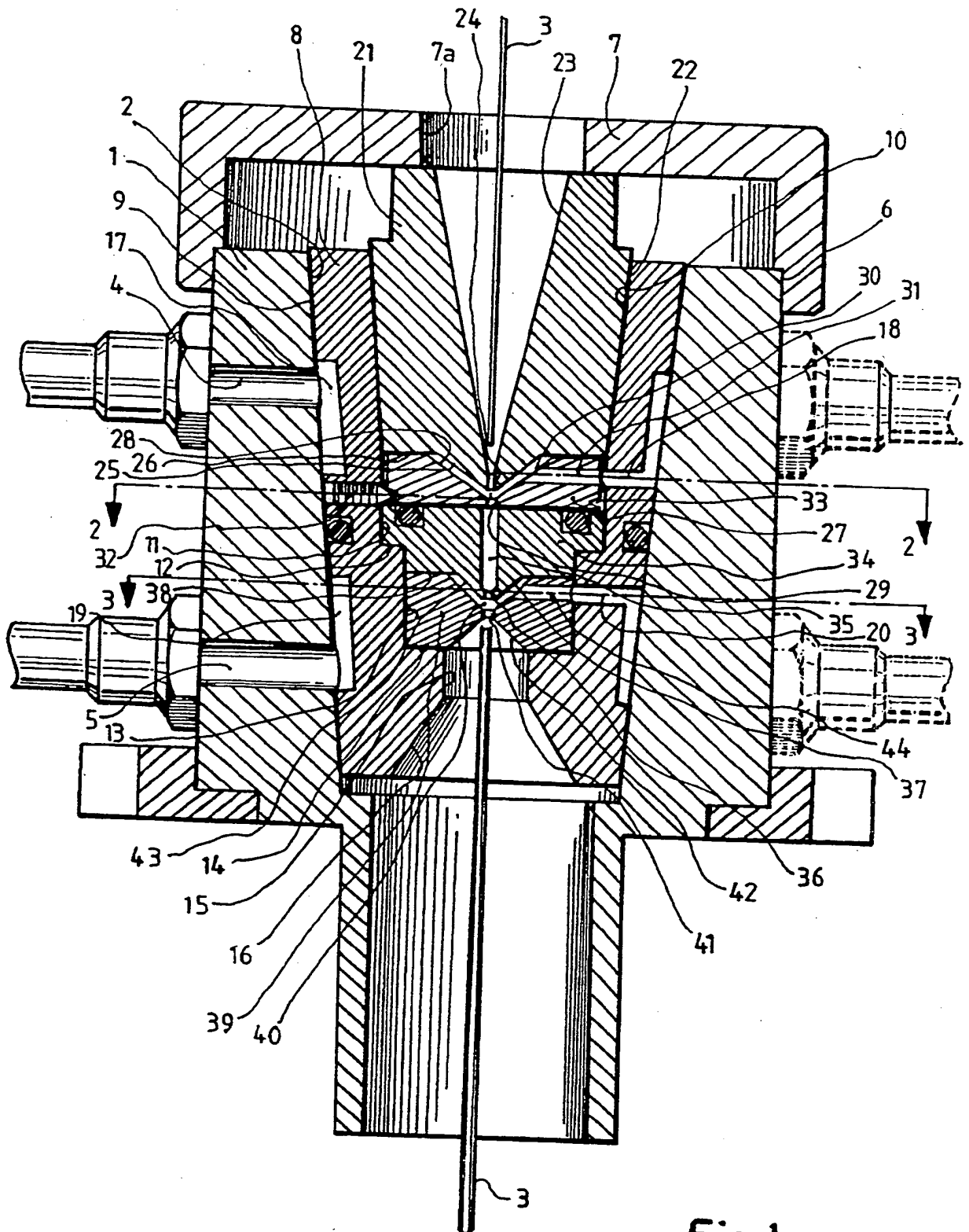


Fig.1

## TECHNICAL FIELD

The present invention relates to a double-coating cup for coating an optic fibre with two protective layers simultaneously, said cup comprising a body having a frusto-conical boring formed axially therein, wherein the largest diameter of said boring lies on the inlet side of the fibre, and a die holder which is pressed firmly in the body and has a frusto-conical outer side surface corresponding to said boring, and further comprising two die nozzles inserted in the die holder for the application of said two layers of coating material.

## BACKGROUND ART

Coating cups used to coat an optical fibre with two coating layers simultaneously are known to the art. These coating cups include radially extending slots or frusto-conical borings through which the coating material is applied on to the fibre. The drawback with these known coating cups is that centering of the fibre in the coating material is unreliable. An additional drawback with such known coating cups is that their construction is often complicated, resulting in handling difficulties when carrying out maintenance work and when cleaning the cups, for example.

## DISCLOSURE OF THE INVENTION

The object of the present invention is to avoid the drawbacks associated with earlier known coating cups and to provide a double-coating cup of simple construction which can be readily dismantled for cleaning or like purposes, and to ensure that the two coating layers will be applied onto the fibre with precision such that the fibre will lie fully concentrically in the protective layers, and that coating of the fibre can be effected so that the fibre is centred in the cup-body without risk of wear through contact with the different die nozzles. This object is achieved with a double-coating cup having the characteristic features set forth in the following Claims.

Thus, an inventive double-coating cup has the advantage of providing a double-coated fibre of very precise diameter and concentricity. Another advantage is that the cup is of simple construction and can be readily handled and maintained.

Other objects of the invention and further advantages afforded by the inventive double-coating cup will be evident from the following description, in which the invention is described in more detail with reference to a preferred exemplifying embodiment thereof and also with reference to the accompanying drawings.

## BRIEF DESCRIPTION OF THE DRAWINGS

Figure 1 is an axial section view of an inventive

double-coating cup taken on the line 1-1 in Figures 2 and 3.

Figure 2 is a cross-sectional view of the double-coating cup taken on the broken line 2-2 in Figure 1.

Figure 3 is another cross-sectional view of the double-coating cup taken on the line 3-3 in Figure 1.

## BEST MODE FOR CARRYING OUT THE INVENTION

The double-coating cup illustrated in Figure 1 includes a body 1 in which there is formed a truncated, conical boring 2 whose largest diameter is located on the inlet side at which a fibre 3 to be coated with two protective layers of coating material is introduced into the cup. The body 1 has two first inlet channels 4, which are diametrically opposed to one another at a first axial distance from said inlet side, and two second inlet channels 5 which are arranged axially spaced from the inlet side and located axially beneath the inlet channels 4. The inlet channels 4 and 5 are connected to pressurized containers containing the two coating materials, by means of nipples and hoses in a manner not shown. On the inlet side, the body is also provided with an externally screw-threaded part 6 for coaction with a holding nut 7 having a central opening 7a for the incoming fibre 3 and for firmly holding the parts of said body 1, in a manner described in more detail here below.

A die holder 8 having a frusto-conical outer surface 9, corresponding to the boring 2, is inserted in the body 1. The die holder 8 is provided with a frusto-conical boring 10 which merges with a first cylindrical boring 11. There then follows a first shoulder 12, a second cylindrical boring 13 whose diameter is smaller than the diameter of the boring 11, a second shoulder 14 and a third, still smaller cylindrical boring 15. This latter boring is followed by a frusto-conical outlet boring 16. The outer surface 9 of the die holder 8 has formed therein first ring-shaped slots 17 which lie opposite the coating material entering through the inlet channels 4 and which form material distribution channels, said slots extending roughly through one-quarter of a circle around the die holder, as illustrated clearly on the right of Figure 2. As will also be seen from Figure 2, the slots 17 connect with first radial borings 18 which open into the junction between the frusto-conical boring 10 and the first cylindrical boring 11. The illustrated embodiment includes four radial borings 18 which are mutually spaced at 90° so as to distribute the coating material uniformly around the fibre, as will be described in more detail herebelow. Provided on the die holder 8 opposite the inlet channels 5 are second ring-shaped slots 19 which form distribution channels for the second coating material and which also extend through roughly one-quarter of a circle around the die holder and connect with second radial borings 20, as

illustrated in Figure 3, these borings 20 opening into the second cylindrical boring 13. These borings 20 are also four in number and are spaced mutually at 90° in order to uniformly distribute the second coating material.

Fitted loosely into the conical boring 10 of the die holder 8 is a fibre-guide die 21. The guide die has an external, frusto-conical surface 22 which corresponds to the shape of the boring 10 and also has a frusto-conical inlet boring 23 which functions to guide the optical fibre into the double-coating cup, this boring 23 merging with a cylindrical guide-boring 24 which opens into a frusto-conical outwardly directed part 25 of the lower end-wall 26 of the guide die.

A first cylindrical die 27 is fitted loosely into the first boring 11 of the die holder. The die 27 has a frusto-conical recess 28 which merges with a cylindrical nozzle-boring 29, and which is arranged so as to form a gap 30 between the recess 28 and the outwardly extending part 25 of the guide die. Radial borings 31 in the die open into the recess 28, and therewith into the gap or slot 30, these borings being disposed opposite the first borings 18 in the die holder, as clearly shown in Figure 2. The borings 31 function to distribute the first coating material uniformly in the gap 30 around the incoming fibre 3, causing, at the same time, the fibre to be centered accurately centred in the die and coated uniformly with coating material. As beforementioned, the die or nozzle 27 is fitted loosely into the die holder 8 and is held and centered in said holder by means of screws 32, the tips of which lie in a circular groove 33 provided in the die.

Located beneath the first die 27 is a cylindrical intermediate die 34 which is press-fitted into the first boring 11 in abutment with the first shoulder 12. The guide die 34 has a cylindrical boring 35 which merges with a narrower guide-boring 36. This guide-boring opens into a frusto-conical outwardly extending part 37 in the bottom end-wall 38 of the guide die.

A second cylindrical die 39 is press-fitted into the second boring 13 of the die holder 8, in abutment with the second shoulder 14. The die has a frusto-conical recess 40 which merges with a cylindrical die-boring 41 and a frusto-conical outlet boring 42. The die 39 is arranged in a manner to form a gap or slot 43 between the outwardly extending part 37 of the intermediate die 34 and the recess 40. Radial borings 44 open into the recess 40, and thus also into the gap 43, in the same manner and for the same reason as that described above with reference to the die 27, as illustrated in Figure 3, so as to distribute the second coating material uniformly around the fibre 3 coated with the first coating material, causing the fibre to be centered in the die and coated uniformly with the second coating material.

The full extension of the fibre 3 through the various die borings has not been shown in Figure 1, for the sake of clarity. The Figure does show,

however, the manner in which a pair of sealing rings is arranged to prevent the throughflow of coating material between the two die nozzles 27 and 39. Thus, one sealing ring is provided in a groove in the nozzle holder 8, between the ring-shaped slots 17 and 19, and one sealing ring is arranged in a groove in the intermediate guide die 34, against the lower end-wall of the first die nozzle 27. The Figure also shows that the body is provided with a cylindrical, downwardly protruding sleeve which surrounds the exiting fibre coated with said two coating materials.

As before mentioned, the coating material used is preferably a UV-curing acrylate, although other materials may be used, of course. The acrylate may be admixed with a UV-hardening paint, so as to incorporate a cable-coding or cable-marking colour in the protective coating. The double-coating cup may also be used to effect a separate colour-coating or colour-marking operation, by drawing a coated fibre once more through the body, the coating material in this case consisting of a paint, preferably a UV-hardening paint, instead of acrylate.

It will be understood that the invention is not restricted to the aforescribed and illustrated embodiment thereof and that modifications can be made within the scope of the following Claims.

#### Claims

1. A double-coating cup for applying two protective coatings simultaneously to an optical fibre, said cup comprising a body provided with an axially extending, frusto-conical boring whose largest diameter is located on the fibre-inlet side, a die holder firmly pressed into the body and having a frusto-conical outer surface which corresponds to the boring, and two die nozzles inserted in the die holder and functioning to apply said two layers of coating material, characterized in that the body (1) has two first diametrically opposed first coating-material inlet channels (4) spaced at a first axial distance from the inlet side, and two second diametrically opposed second coating-material inlet channels (5) spaced at a second axial distance from said inlet side; in that the die holder (8) has a frusto-conical boring (10) whose largest diameter lies on the fibre inlet-side, a first cylindrical boring (11) which is a continuation of the conical boring, a first shoulder (12), a second, smaller cylindrical boring (13), a second shoulder (14), and a third, smallest cylindrical boring (15); in that the outer surface (9) of the die holder has first ring-shaped slots (17) located opposite the first inlet channels (4) and first radial borings (18) which connect with said ring-shaped slots (17) and which open into the junction between the frusto-conical boring (10) and the first cylindrical

boring (11), and second ring-shaped slots (19) which are located opposite the second inlet channels (15) and which connect with second radial borings (20) which open into the second cylindrical boring (13); in that a guide die (21) having a frusto-conical outer surface (22) is inserted into the conical boring (10) of the die holder and includes a frusto-conical inlet boring (23) which merges with a cylindrical guide boring (24) in a frusto-conical outwardly projecting part (25) in the end-wall (26) of said die; in that a first cylindrical die nozzle (27) having a frusto-conical recess (28) which merges with a cylindrical nozzle boring (29) is inserted in the first cylindrical boring (11) in a manner such as to form a first coating-material gap or slot (30) between the die nozzle (27) and the guide die (21), said die nozzle being provided with distribution channels (31) which connect with the first radial borings (18) and which function to distribute said coating material around the fibre and therewith centre the fibre in the nozzle; in that a cylindrical intermediate guide die (34) provided with a cylindrical boring (35) is positioned against the first shoulder (12) and has a frusto-conical outwardly projecting part (37) in the end-wall (38) of said die; and in that a second cylindrical die nozzle (39) having a frusto-conical recess (40) which merges with a cylindrical nozzle boring (41) is positioned in the second cylindrical boring (13) and in abutment with said second shoulder (14) in a manner such as to form a second coating-material gap or slot (43) between the die nozzle (39) and the intermediate guide die (34), said die nozzle having distribution channels (44) which connect with the second radial borings (20) and which distribute coating material around the fibre coated with said first coating material and therewith centre the fibre in said die nozzle.

2. A cup according to Claim 1, characterized in that the distribution channels (31, 44) in the die nozzles (27, 39) are formed by circumferentially disposed and uniformly spaced radial borings which open into the frusto-conical recesses (28, 40).

3. A cup according to Claim 2, characterized in that the intermediate guide die (34) is provided with an axially disposed sealing ring against the first die nozzle (27); and in that a sealing ring is mounted on the outer surface (9) of the die holder (8) between the first and the second radial borings.

4. A cup according to Claim 3, characterized in that the first die nozzle (27) is loosely fitted into the first cylindrical boring (11) and is firmly held and centered in the die holder by means of screws

(32).

5. A cup according to Claim 4, characterized in that the intermediate guide die (34) is press-fitted in the first boring (11); and in that the second die nozzle (39) is press-fitted in the second cylindrical boring (13).

6. A cup according to any one of the preceding Claims, characterized in that the body (1) is provided on the inlet side with a screw thread (6) for coaction with a holding nut (7) provided with an opening (7a) and lying in abutment with the guide die (21).

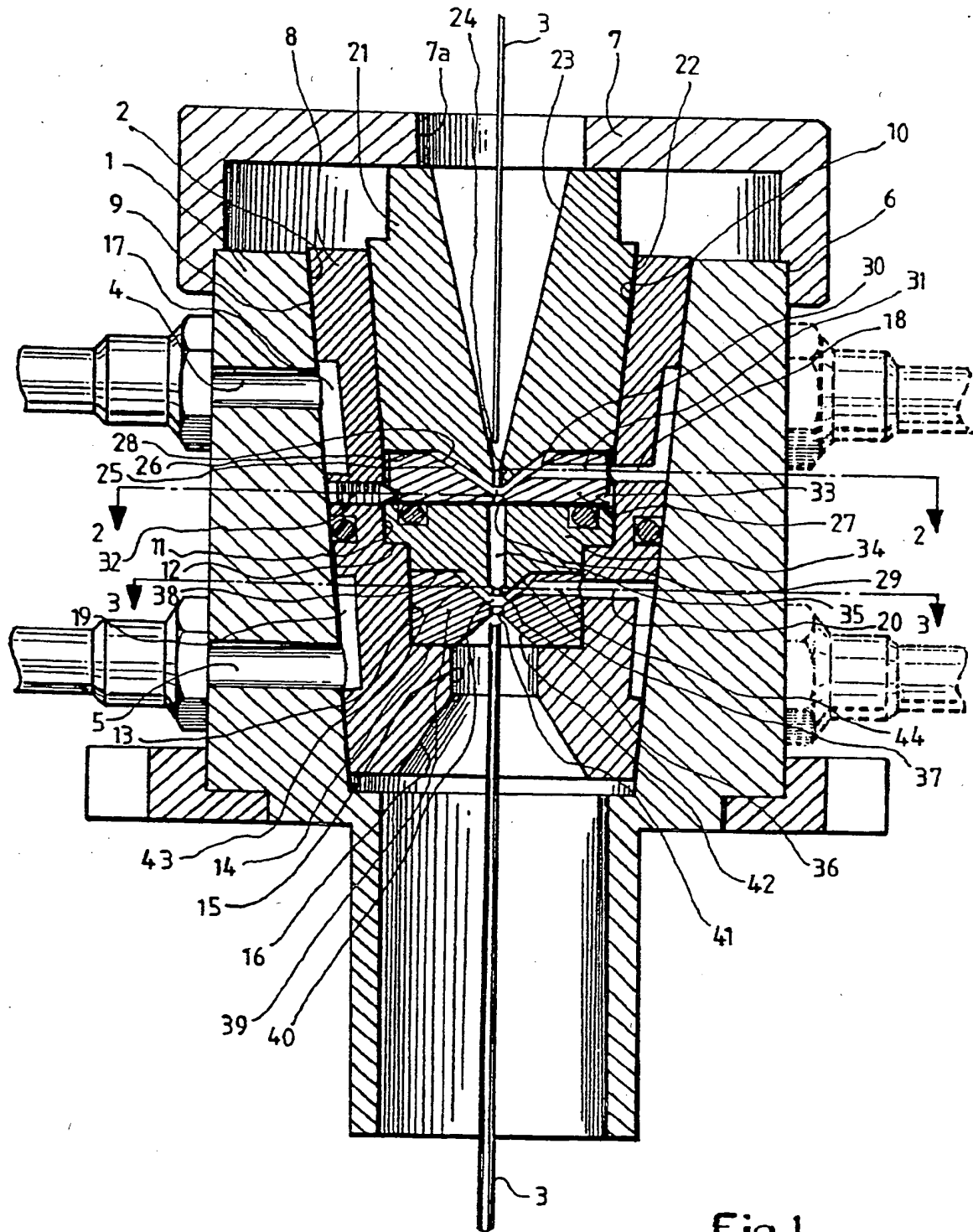
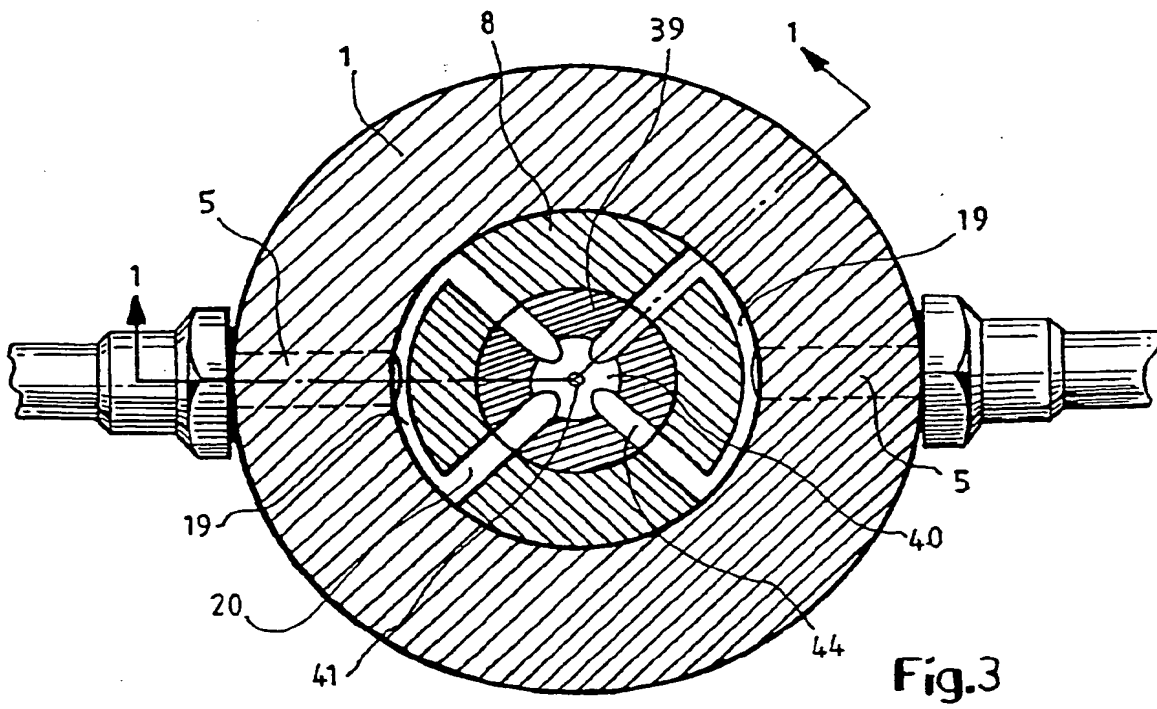
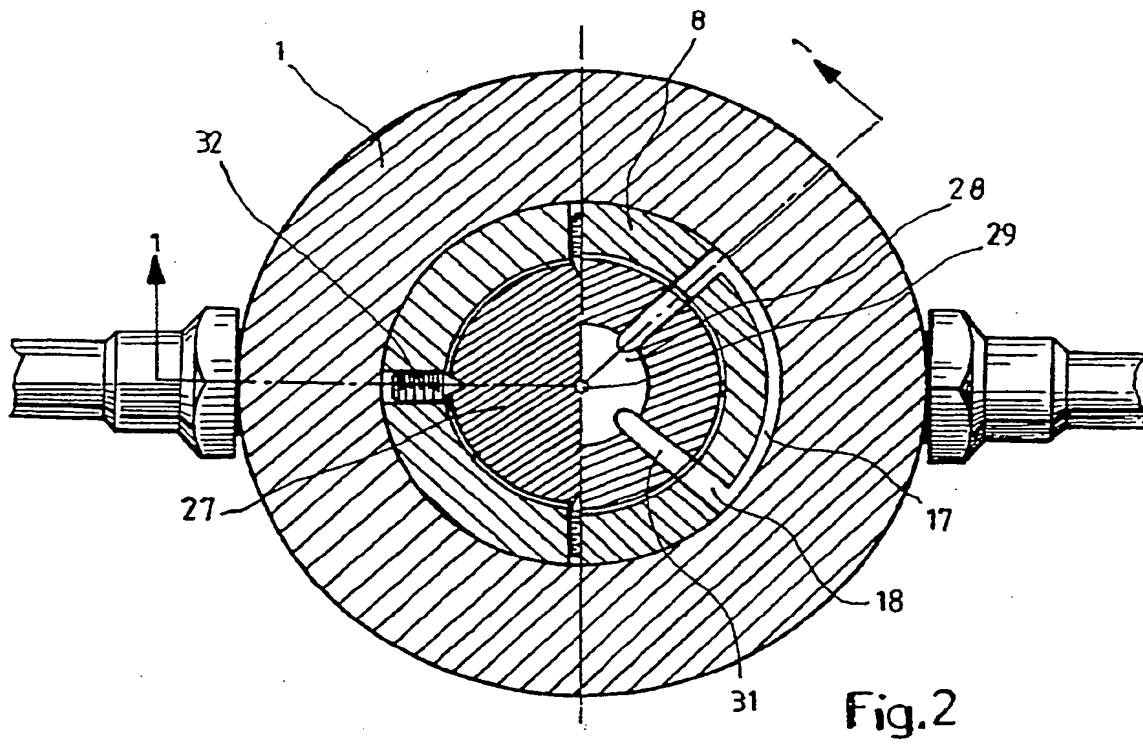


Fig.1







European Patent  
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# EUROPEAN SEARCH REPORT

Application number  
91850135.4

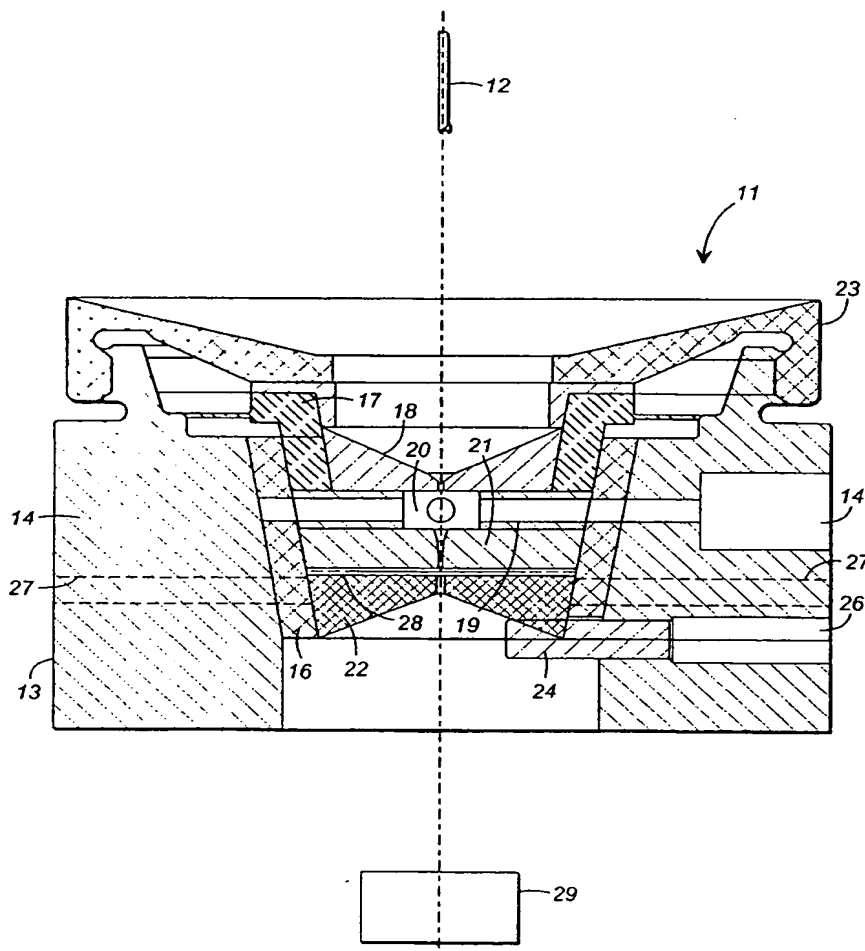
DOCUMENTS CONSIDERED TO BE RELEVANT			
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl.)
A	EP-A1-0 114 346 (WESTERN ELECTRIC COMPANY, INCORPORATED)	1-6	C 03 C 25/02 B 29 C 47/02
A	EP-A2-0 306 329 (AMERICAN TELEPHONE AND TELEGRAPH COMPANY)	1-6	
			TECHNICAL FIELDS SEARCHED (Int. Cl.)
			B 05 C B 29 C B 29 F C 03 C
The present search report has been drawn up for all claims			
Place of search STOCKHOLM		Date of completion of the search 09-09-1991	Examiner HALLNE M.
<p><b>CATEGORY OF CITED DOCUMENTS</b></p> <p>X : particularly relevant if taken alone  Y : particularly relevant if combined with another document of the same category  A : technological background  O : non-written disclosure  P : intermediate document</p> <p>T : theory or principle underlying the invention  E : earlier patent document, but published on, or after the filing date  D : document cited in the application  L : document cited for other reasons  &amp; : member of the same patent family, corresponding document</p>			

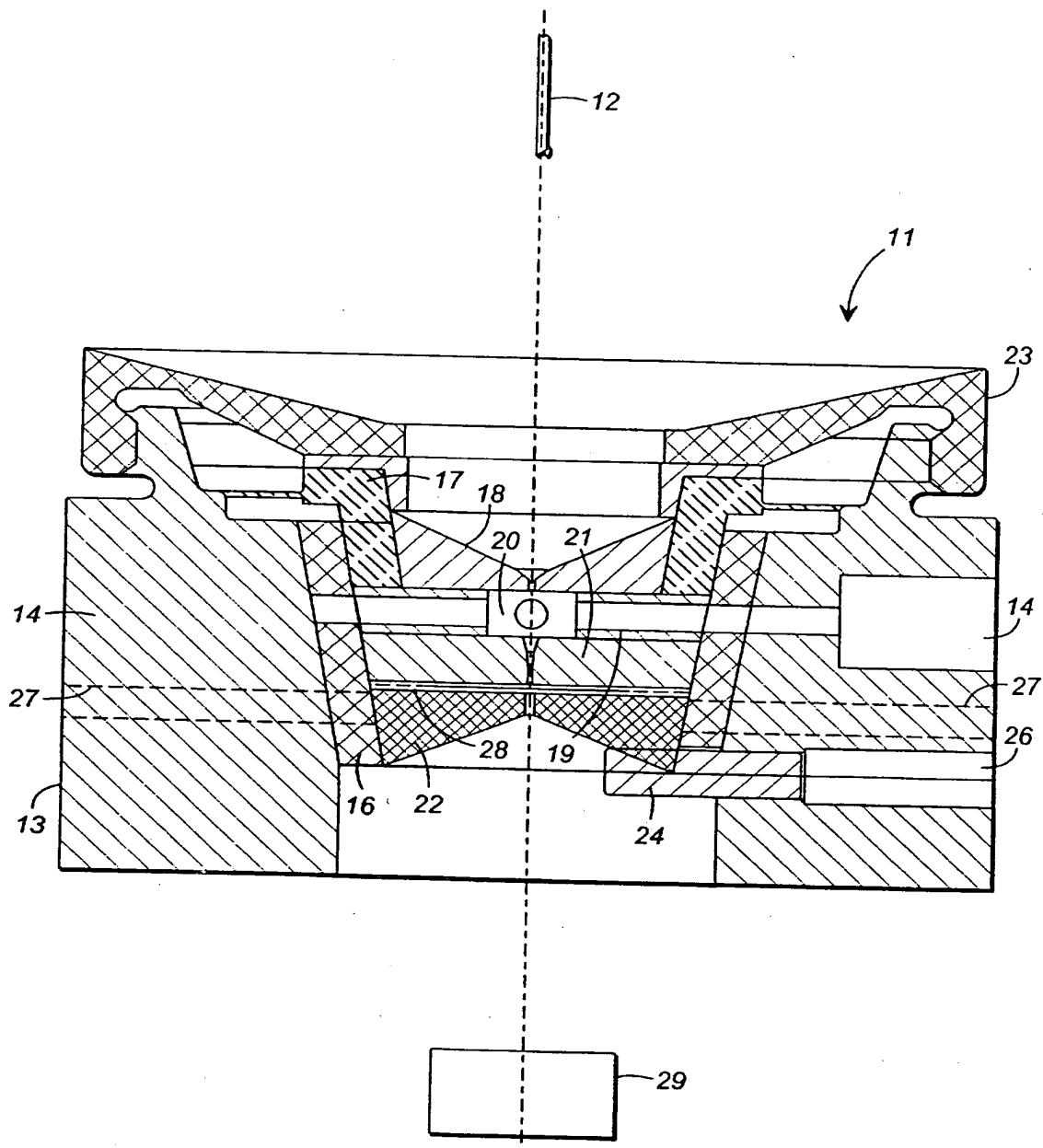


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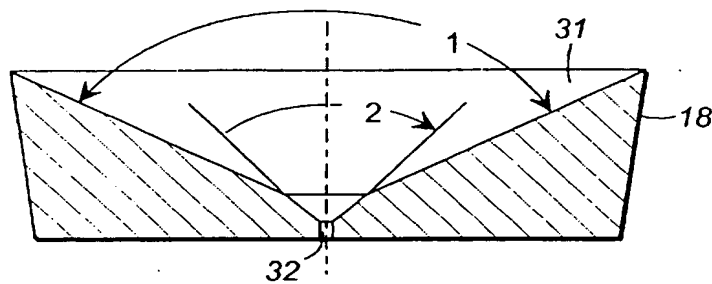
**United States Patent** [19][11] **Patent Number:** **6,030,664****DiMarcello et al.**[45] **Date of Patent:** **Feb. 29, 2000**[54] **BICONIC COATING DIE FOR MAKING COATED OPTICAL FIBERS**[75] **Inventors:** **Frank V. DiMarcello**, Annandale;  
**Arthur C. Hart**, Chester; **Richard G. Huff**, Basking Ridge; **Karen S. Kranz**, Middlesex, all of N.J.; **Lisa M. Larsen-Moss**, Stone Mountain, Ga.[73] **Assignee:** **Lucent Technologies Inc.**, Murray Hill, N.J.[21] **Appl. No.:** **09/116,548**[22] **Filed:** **Jul. 15, 1998**[51] **Int. Cl.<sup>7</sup>** ..... **B05D 1/18; B05D 1/36; B05D 5/06; B05C 3/12**[52] **U.S. Cl.** ..... **427/402; 427/163.2; 427/389.8; 427/407.3; 427/434.7; 118/405; 118/420; 65/447; 65/529**[58] **Field of Search** ..... **427/163.2, 434.7, 427.402, 407.3, 389.8; 118/405, 420; 65/447, 529**[56] **References Cited****U.S. PATENT DOCUMENTS**4,704,307 11/1987 Jochem et al. .... 427/163.2  
5,127,361 7/1992 Matsuda et al. .... 118/405*Primary Examiner*—Katherine A. Bareford[57] **ABSTRACT**

A method and apparatus for high velocity dual coating optical fibers includes passing the fiber to be coated through an entrance die into a coating reservoir, to which liquid coating material is supplied under pressure. The diameter of the die passage and the pressure of the fluid is sufficient to prevent air from entering the reservoir. The coated fiber is passed through a centering and sizing die wherein the recirculation of excess coating material is controlled to produce centering forces on the fiber at the high fiber velocity, and wherein the initial coating is sized. The fiber is then passed through a second coating reservoir into an exit die which sizes the second coating. Upon leaving the exit die, the coatings are cured.

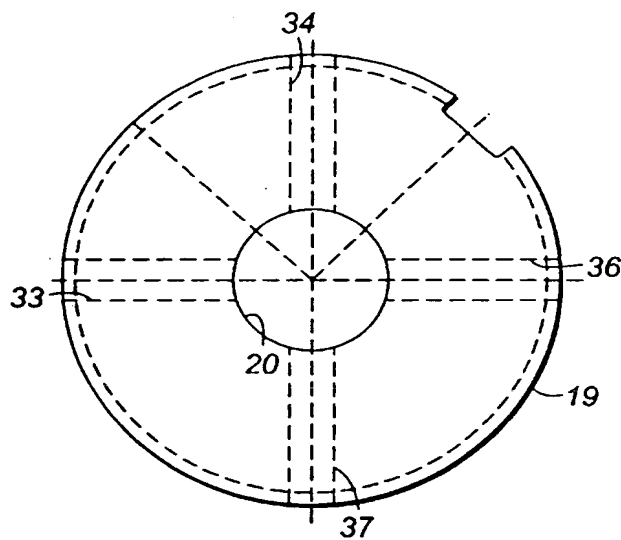
**12 Claims, 4 Drawing Sheets**



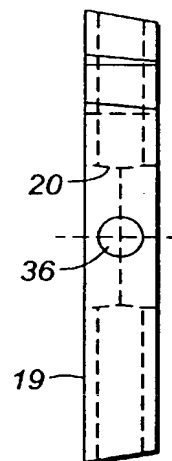
**FIG. 1**



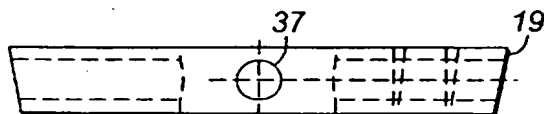
**FIG. 2**



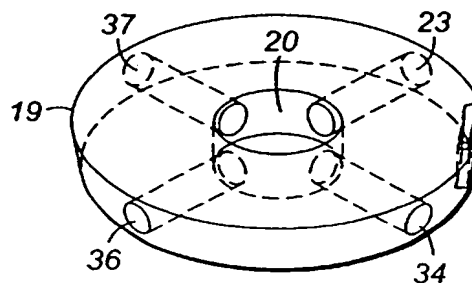
**FIG. 3a**



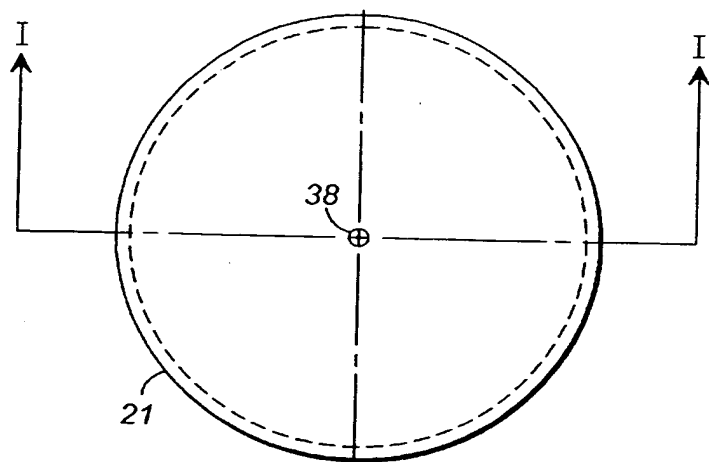
**FIG. 3c**



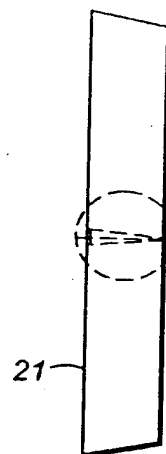
**FIG. 3b**



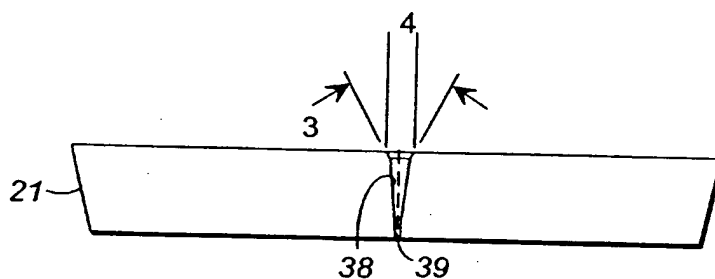
**FIG. 3d**



**FIG. 4a**

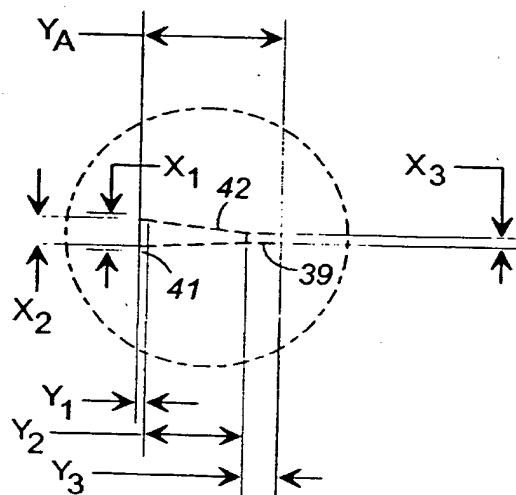


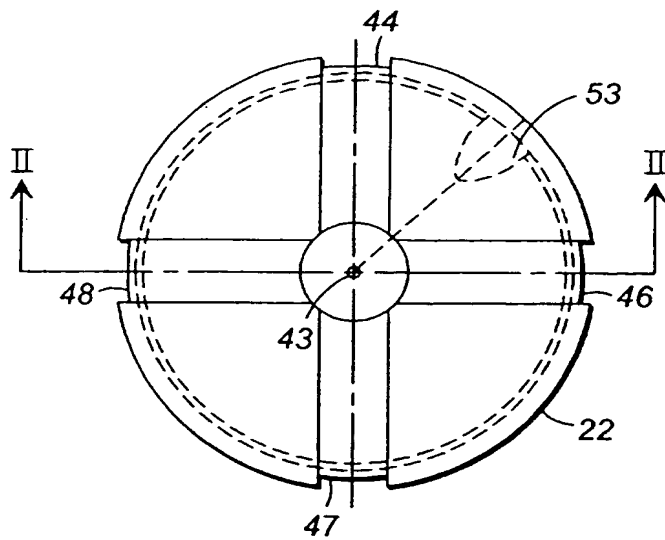
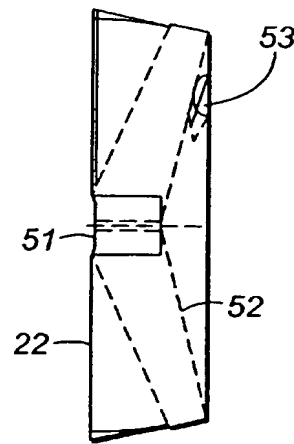
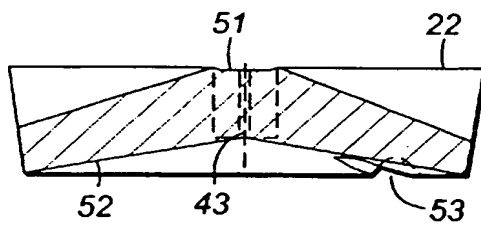
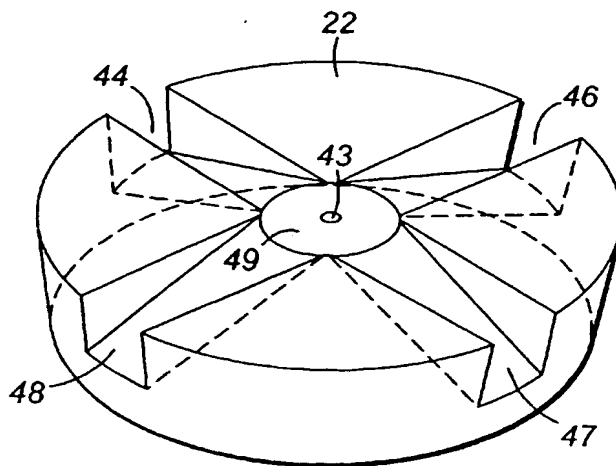
**FIG. 4c**



**FIG. 4b**

**FIG. 4d**



**FIG. 5a****FIG. 5c****FIG. 5b****FIG. 5d**

# BICONIC COATING DIE FOR MAKING COATED OPTICAL FIBERS

## FIELD OF THE INVENTION

This invention relates to coating optical fibers and, more particularly, to an apparatus for applying multiple coatings at high speeds.

## BACKGROUND OF THE INVENTION

Optical fiber production generally involves drawing the fiber, which usually is of silica glass, and then applying a dual layer of coating materials to the fiber. A first layer typically comprises a relatively soft material and the second layer typically comprises higher modulus curable polymeric material for maintaining high strength and abrasion resistance. Each fiber thus coated must be capable of withstanding, over its entire length, a maximum stress level to which the fiber will be exposed during installation and service. A single fiber failure can result in the loss of several hundreds of circuits. The coating, dual or otherwise, should function to prevent airborne particles from impinging upon and adhering to the surface of the drawn fiber, which could weaken it or even affect its transmission properties. Also the coating shields the fibers from surface abrasion, which could occur as a result of subsequent manufacturing processes and also handling during installation.

Optical fibers are usually coated during a wet coating process which typically involves drawing the fiber through a reservoir of liquid polymer material and then curing the liquid polymer to harden it by exposure to curing radiation, such as, for example, ultra-violet light. In the dual coating process, the coatings are applied in tandem or simultaneously (within the same applicator or die assembly). The tandem arrangement applies a first coating layer which is then cured, and then the second coating layer is applied and cured. In the simultaneous dual coating arrangement, both coats are applied after which they are cured. In both cases, the primary coating is typically a low modulus material and the second coating is a relatively high modulus material. During the wet coating process, air bubbles may become entrained between the fiber and the first or primary and secondary layers, or within the actual layers themselves. An air coating interface can become unstable at higher speeds which leads to the formation of bubbles. Such bubbles give rise to a number of problems. Bubbles can cause losses in signal transmission by, for example, causing inhomogeneity of the modulus near the glass surface which can cause mechanical distortion of the fiber. In addition, bubbles can weaken the mechanical strength of the coated fiber.

There has been, and continues to be, increasing emphasis on fiber waveguide draw speeds. Much effort has been expended on increasing fiber velocity in the coating process while avoiding the formation of bubbles in the coating layers. In U.S. Pat. No. 4,246,299 of Ohls, a fiber is passed through an applicator having a die body that defines a small, vertically oriented, longitudinally tapered passage having a reservoir disposed about it. A series of radial ports provide fluid communication between the reservoir and the passage. Turbulence within the coating material, which causes entrapment of air bubbles, is reduced by maintaining the level of coating material in the passage. In U.S. Pat. No. 4,374,161 of Geyling et al. there is shown a coating arrangement wherein the fluid coating material is directed radially toward the fiber. The passage diameter for the fiber is large enough to prevent contact with the fiber, while the pressure of the fluid coating material is high enough to substantially

prevent air from entering the applicator. In U.S. Pat. No. 4,480,898 of Taylor, there is shown a dual coating applicator having a die that provides for formation of a gap between the die and the first coated layer. A second die is located at the exit of the first die, with the second coating material flowing through a narrow passage between the first and second dies. The second die also provides for a gap so that the second layer is applied at a free surface at the point of contact with the first layer. This arrangement has been found to eliminate instabilities and coating non-uniformities at increased speeds.

Despite the numerous arrangements for reducing turbulence at high fiber velocities, it has, thus far, been extremely difficult to achieve uniform and consistent results with fiber velocities greater than 14 m/sec. In addition, the coating operation itself, in the foregoing arrangements, requires an operator to monitor and continuously adjust fluid pressure, and to adjust orientation of the die, for example, to insure coating concentricity. Thus, the coating processes of the prior art are susceptible to a great deal of improvement.

## SUMMARY OF THE INVENTION

The present invention, in a preferred embodiment thereof, is a dual coating applicator for an optical fiber which eliminates or overcomes many of the problems present in prior art arrangements, and which produces consistent results at fiber velocities in excess of 20 m/sec and as great as 33 m/sec, a rate that has not been approached thus far in the prior art.

The applicator of the invention uses a smaller entry die with no coating set point above the entry die, thereby eliminating the need for operator pressure control, the applicator has increased fiber centering forces for improved process stability and prevents wandering of the fiber in the die, and which reduces coating concentricity sensitivity to die position and tilt, thereby reducing the need for operator adjustments.

In greater detail, the applicator of the invention has a bi-conic entry port having a minimum diameter within a range of  $200\mu-400\mu$ , which opens into a primary coating fluid reservoir into which the fluid coating under pressure is applied through multiple ports. A high pressure gradient is then created halfway to the entry port passage and the reservoir which inhibits fluid flow into the passage. A primary coating die having a bi-conic passage therethrough is located at the reservoir exit and functions to size and center the primary coating on the fiber. As the fiber passes through the first biconic die passage, the high pressure gradient prevents bubble incorporation in the coating. The coating recirculation in the primary coating fluid reservoir can act to center the fiber and prevent it from touching the walls of the entry die, providing the recirculation pattern is properly formed to generate sufficient centering forces. However, a conic angle of approximately 12 degrees leading into the primary coating die generates the major centering forces for the applicator. The minimum diameter of the passage is also approximately  $200\mu-400\mu$ , depending upon the desired coated fiber diameter, the coating material, and the fiber velocity. A first conic angle of approximately  $60^\circ$  and a second conic angle of approximately  $12^\circ$  leading into a passage diameter of approximately 9 mils produces an almost ideal recirculation pattern at high speeds for generating the desired centering forces, and the coated fiber emerging from the die is concentric, with a maximum variation in fiber diameter of  $1-2\mu$ . The bi-conic passage opens into a second reservoir having the secondary fluid



coating under pressure therein, and the fiber exits through an exit die having a passage diameter of approximately 500  $\mu$ , where a final sizing and centering occurs. This final diameter depends upon the desired coated fiber diameter, the coating material, and the fiber velocity. As will be apparent hereinafter, the angles of the bi-conic passages as well as their diameters, are important for achieving the desired result. After emergence from the exit die, the coatings are cured by suitable means.

The basic principles of the invention, as well as the features thereof, are equally applicable to both dual and tandem processing, as will be apparent from the following detailed description, read in conjunction with the drawings.

### DESCRIPTION OF THE DRAWINGS

FIG. 1 is an elevation view in cross-section of a dual coating applicator in accordance with the present invention;

FIG. 2 is a cross-sectional view of the entrance die of the applicator of FIG. 1;

FIGS. 3a, 3b, 3c and 3d are various views of the cavity member of the applicator of FIG. 1;

FIGS. 4a, 4b, 4c and 4d are various views of the primary coating die of the applicator of FIG. 1; and

FIGS. 5a, 5b, 5c and 5d are various views of the exit die of the applicator of FIG. 1.

### DETAILED DESCRIPTION

The following description is directed to a method and apparatus for dual coating of optical fibers, at speeds or fiber velocity hereto considered unachievable, and wherein the coatings are of good quality, being substantially bubble free and concentric within acceptable tolerances. It is to be understood that the method of application and the applicator itself are adaptable for use in coating other filamentary materials besides the silica glass generally used in optical fibers.

In FIG. 1 there is shown, in cross-section, a dual coating applicator 11 embodying the principles and features of the present invention. For clarity, the fiber 12 to be coated has not been shown passing through the applicator 11, but it is to be understood that it does so pass along the centerline in the direction of the arrow, as shown. Applicator 11 comprises a block 13 having ports 14 and 27 (shown in dashed lines) therein for the radial introduction of coating material as will be discussed hereinafter and further having tapered sleeves 16 and 17 for holding an entrance die 18, a primary die cavity member 19 which forms a fluid coating reservoir 20, a middle or primary die 21 and an exit die 22. A retaining cap 23 functions to apply pressure on the tapered members and on the dies, which, as shown, are also tapered, to retain them in place. In addition, a locating pin 24, insertable through bore 26 in block 13 functions to circumferentially locate the die assembly, as will be discussed hereinafter. As is shown in FIG. 1, a secondary coating cavity 28 is formed between the exit end of primary die 21 and the entrance end of exit die 22.

All three dies 18, 21, and 22 have central passages therein which, to avoid confusion, are not numbered in FIG. 1. These passages are critical and will be discussed in greater detail hereinafter. In the operation of applicator 11, which is a simultaneous dual coating applicator, the fiber 12 is threaded through the passage in entrance die 18 and is drawn down through primary cavity member 19, through the passage in primary die 21, through secondary cavity 28 and through the passage in exit die 22. After emerging from exit

die 22, the coated fiber is passed through a curing station 29 which may cure the coatings with ultraviolet radiation, heat, or whatever curing radiation is appropriate for the particular coating materials used. Typical of primary or inner coating materials are soft low modulus materials such as silicone, hot melt wax, or any of a number of acrylic or polymeric materials having a relatively low modulus. Typical materials for the second or outer coating are high modulus (3000-5000 cp) polymeric or acrylics. In practice in a simultaneous applicator as shown in FIG. 1, both materials may be low and high modulus acrylic.

In greater detail, in the coating process of the invention using the applicator 11, the fiber 12 passes through entrance die 18 into the reservoir 20 where the primary or inner coating is contained under pressure. The diameter of the passage in the entrance die is chosen such that it is sufficient to avoid fiber impingement thereon and sufficiently narrow that a high pressure gradient is created such that the liquid coating under pressure in reservoir 20, instead of rising up through the passage to the top where it encounters air, and hence entrains air bubbles is effectively blocked. Thus, air is prevented from entering the reservoir, thereby relieving the operator of continuously monitoring the fluid pressure. It is known to then seal the reservoir from the air, as is shown in U.S. Pat. 4,374,161 of Geyling et al. However, the Geyling et al. apparatus for accomplishing such a result is considerably more complicated than the present arrangement and is not, apparently, amenable to extremely high velocity fiber coating, as is the present arrangement. A coating pressure of 150-200 psi for a passage diameter approximately twice that of the fiber to be coated has been found to be sufficient to eliminate bubbles at fiber velocities in the range of 25 m/sec to 35 m/sec. The now coated fiber passes from the reservoir 20 into primary die 21 which has a passage therethrough having a bi-conic section and a straight section. The entrance angle of the passage, i.e., the first conic section, is approximately 60° and that of the second conic section is approximately 12°. The second section connects to a straight passage of approximately 0.009 inches diameter. At the velocities of operation, this combination of an entrance angle of 60° and a small second angle, 12°, and a small but sufficient diameter passage of sufficient length function to generate centering forces for the coating and the fiber as well as sizing forces due to the passage diameter. The fiber, as it enters the die, has an excess amount of coating thereon, which, as the fiber passage through to decreasing diameter of the conic section is, in effect, scraped off of the coated fiber and tends to recirculate back toward the reservoir 20. This movement of the excess coating material is shaped by the narrow, small angle conic section into exerting radially uniform centering forces on the coated fiber as it passes through into the straight portion of the passage, where it is sized. The bi-conic configuration of the passage in the primary die 21 insures that there is less than one micron (1 $\mu$ ) of variation in the coated fiber diameter, whereas, without the bi-conic configurations, variations of 4 to 5 microns may occur. The narrow angle of the conic section (12°) as opposed to a prior art angle of 24° or more and its length shape the recirculating flow of the excess coating to insure proper centering.

The secondary coating in the cavity or reservoir 28 is under a pressure of 50-250 psi which further functions to seal off the die passages from the air. The passage in the exit die performs a sizing operation on the second or outer coating so that the coated fiber emerges from the applicator 11 with centered coatings of proper or desired diameter.

FIGS. 2 through 5 depict various components of applicator 11 in greater detail.

FIG. 2 is a cross-sectional view of entrance die 18, which is preferably made of hardened steel, such as, for example, tool steel. Die 18 has a bi-conic opening 31 having a first cone angle  $\phi_1$ , leading to a second cone angle  $\phi_2$ , which in turn leads to a straight passage 32. The angle  $\phi_1$  is in a range of 125°–130°, while the angle  $\phi_2$  is approximately 90°. These angles or sloped surfaces function primarily to guide the fiber as it is first being threaded into passage 32, hence they can vary somewhat from the angles given. Passage 32 is, on the other hand, more critical as to its diameter. It has been ascertained that a diameter of 0.009–0.010 inches functions to allow passage of fiber 12 without striking the walls of the passage, while also preventing flow of the pressurized coating material to the top thereof where it can contact the air.

FIGS. 3a, 3b, 3c, and 3d are different views of the primary cavity member 19, preferably made of hardened steel and which, as can be seen, has four fluid passage bores 33, 34, 36, and 37 which communicate with bores 14 in block 13, and which lead to the center cavity or reservoir 20, into which the liquid coating is delivered under pressure. FIG. 3d is a perspective view of member 19 showing the relationship of bores 33, 34, 36, and 37 to reservoir 20.

FIGS. 4a, 4b, 4c, and 4d are different views of the primary die 21, wherein FIG. 4b is a cross-sectional elevation view along the line I—I of FIG. 4a, and FIG. 4d is a detail taken from the circled portion of FIG. 4c. Die 21, which is also preferably made of hardened steel, has a central bi-conic passage 38 opening into a straight passage 39. Bi-conic passage has a first conic section 41 at an angle  $\phi_3$  and a second conic section 42 at an angle  $\phi_4$ . Angle  $\phi_3$  can be approximately 60° however, angle  $\phi_4$  is critical in forming or shaping the fluid recirculation to produce the desired centering forces. In practice, at fiber velocities in the 25 m/sec–35 m/sec range, an angle  $\phi_4$  of 12° has been found to be optimum. This angle  $\phi_4$  can vary slightly depending upon fiber diameter, velocity and the materials used, and is much smaller than what has heretofore been used in the prior art, where it is typically in the neighborhood of 24°. Such a large angle has proven to be unsuitable for high fiber velocities. FIG. 4d depicts the details and dimensional parameters of the passage through die 21 in an embodiment that has been found to yield the desired results as discussed hereinbefore. Conic section 41 has an entrance diameter  $X_1$  and a depth  $Y_1$ , section 42 has an entrance diameter  $X_2$  and a depth (or length)  $Y_2$  and straight section 39 has a depth or length  $Y_3$  and a diameter  $X_3$ . Member 21 has a thickness  $Y_4$ .

FIGS. 5a through 5d are various views of the exit die 22, with FIG. 5b being a cross-section thereof along the line II–II of FIG. 5a, and FIG. 5d being a perspective view thereof. Die 22 has a straight central fiber passage 43 therethrough which functions to size the second coating. Thus, the diameter of passage 43 is proportional to the desired outer diameter of the dual coated fiber, and hence, may vary with that diameter. In one embodiment, the diameter of passage 43 is 0.2. Passage 43 may also have, as an entrance portion to passage 43, a conic section (not shown) for accommodating excess fluid coating material. The angle of this conic section may be, for example, approximately 24°, with an entrance diameter of approximately 0.0445 in. Die 22, which is preferably of hardened steel, has four sloped fluid coating passages 44, 46, 47, and 48 which conduct the fluid under pressure to the top of a central hub portion 49 which has a depressed portion 51 which forms fluid reservoir 28 between dies 21 and 22, as seen in FIG. 1. The underside 52 of die 22 has a curved locator notch 53 into which pin 24, shown in FIG. 1, fits to

locate passages 44, 46, 47, and 48 relative to bores 27, shown in FIG. 1.

In one example of the applicator of FIG. 1,  $\phi_1$  was 127°,  $\phi_2$  was 90°, the diameter of passage 32 was 0.0090 in.,  $\phi_3$  was 60°,  $\phi_4$  was 12°,  $X_1$  was 0.0362 in.,  $X_2$  was 0.0270 in.,  $X_3$  was 0.009 in.,  $Y_1$  was 0.0080 in.,  $Y_2$  was 0.0856 in.,  $Y_3$  was 0.0314 in., and  $Y_4$  was 0.1250 in. With a fluid pressure of approximately 150 psi, excellent results were achieved at fiber velocities in excess of 25 m/sec.

The principles of the present invention are equally applicable to a tandem coating process, not shown. Tandem coating has the advantage of allowing inspection after application and curing of the first coat before the second coat is applied. A tandem coating arrangement would allow inspection and curing after the fiber emerges from primary die 21, after which the fiber would be passed through die 22 or an equivalent.

In concluding the detailed description, it should be noted that it will be obvious to those skilled in the art that many variations and modifications may be made to the preferred embodiment without substantial departure from the principles of the present invention. All such variations and modifications are intended to be included herein as being within the scope of the present invention, as set forth in the claims. In the claims hereafter, the corresponding structure, materials, acts and equivalents of all means or step plus function elements are intended to include any structure, material, or acts for performing the functions with other claimed elements as specifically set forth.

We claim:

1. A fiber coating process comprising passing a fiber at a velocity in excess of twenty meters per second through an entrance die bi-conic passage into a coating fluid reservoir; supplying coating fluid to the reservoir at a pressure such that bubbles are prevented from entering the coating fluid; passing the fiber through a second die passage having a bi-conic section and a straight section having an exit end while controlling the re-circulation of excess coating material to generate centering forces within the bi-conic section of the passage, and thereafter sizing the coated fiber in the straight section prior to curing the coating; and curing the coating.
2. A fiber coating process as claimed in claim 1 and further including the steps of applying and sizing a second coat on the fiber by passing the fiber through a second reservoir immediately adjacent the exit end of the straight section of the second die passage; supplying the second coating material under pressure to the second reservoir; and a third die having a passage having a diameter sufficient to size the dual coated fiber.
3. A fiber coating process as claimed in claim 2 wherein the step of passing the fiber through the second reservoir and the third die precedes the step of curing the coating.
4. A fiber coating process as claimed in claim 2 wherein the pressure of the second coating material is in the range of 50–250 psi.
5. A fiber coating process as claimed in claim 1 wherein the linear velocity of the fiber being coated is in the range of 25–35 meters per second.
6. A fiber coating process as claimed in claim 1 wherein the pressure of the coating fluid supplied to the reservoir is in the range of 150–200 psi.
7. An applicator assembly for applying a coating to a fiber at a fiber velocity equal to or greater than 25 meters per second, said applicator comprising:

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an entrance die having a passage therethrough having a bi-conic section and a straight section having an exit end, the diameter of said straight section being sufficient to avoid fiber impingement thereon and sufficiently small to prevent coating fluid under pressure excessively rising therein;

a coating fluid reservoir adjacent said exit end of said straight section;

means for applying fluid coating material under pressure to said reservoir; and

a primary die adjacent said fluid reservoir and having an entrance end and an exit end, said primary die having a fiber passage therethrough having in sequence a bi-conic section of a relatively wide entrance angle and a narrow angled conic section and a straight section, the angle of said narrow angled conic section being chosen to create fiber centering forces within said fiber passage and said straight section having an exit end and having a diameter sufficient to size the coated fibers.

8. An applicator assembly as claimed in claim 7 and further comprising an exit die having a passage therethrough;

means including said exit die and the exit end of said primary die for forming a second coating fluid reservoir;

means for applying fluid coating under pressure to said second reservoir; and

said passage through said exit die having a diameter sufficient for sizing a second coating on the fiber.

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9. An applicator assembly for applying a coating to a fiber at a fiber velocity greater than 20 meters per second, said applicator comprising:

an entrance die having a passage therethrough having a conic section and a straight section having an exit end, the diameter of said straight section being sufficient to avoid fiber impingement thereon and sufficiently small to prevent coating fluid under pressure excessively rising therein;

a coating fluid reservoir adjacent said exit end of said straight section;

a primary die adjacent said fluid reservoir, said primary die having a fiber passage therethrough having, in sequence, a bi-conic section of a relatively wide entrance angle and a narrow angled conic section and a straight section, the angle of said narrow angled conic section being approximately  $12^\circ$  to create fiber centering forces within said fiber passage and said straight section having an exit end and having a diameter sufficient to size the coated fibers.

10. An applicator assembly as claimed in claim 9 wherein the length of said narrow angled conic section is approximately 0.0856 inches.

11. An applicator assembly as claimed in claim 10 wherein the diameter of said straight section of said fiber passage is approximately 0.009 inches.

12. An applicator assembly as claimed in claim 11 wherein the diameter of said passage in said entrance die is 0.009–0.010 inches.

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